EXPERIMENTS ON EROSION AND FLUIDIZATION
STRENGTH OF KAOLINITE CLAY

By
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To the Faculty of Washington State University:

The members of the Committee appointed to examine the dissertation of ADAM RICHARD MAXWELL find it satisfactory and recommend that it be accepted.

__________________________
Chair

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EXPERIMENTS ON EROSION AND FLUIDIZATION
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Abstract

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The present dissertation is comprised of three manuscripts. In the first part, the self-weight fluidization behavior of kaolinite clay was examined via batch sedimentation tests; two of the experimental cases were also simulated numerically. It was found that mixing conditions and gas entrainment can have significant effects on the reproducibility of batch sedimentation experiments. Further, it was shown that traditional methods of detecting self-weight fluidization via changes in the batch curve or flux curve are not effective for the cases examined. Numerical experiments showed close agreement with the overall conditions observed, further bearing out the assertion that these traditional methods are not sensitive to fluidization. The final sedimented conditions for these tests were used to determine the experimental conditions for the remaining parts. The second part considers the fluvial erosion strength of kaolinite clay, when subjected to water flow in flume experiments. The effects of sediment age, clay content, water content, and microbes are examined. The
erosional strength of sediment was shown to increase with sediment age, due
to thixotropic hardening. This may occur over a longer time period than previ-
ously thought. Erosional strength and water content were shown to vary in an
inverse manner, and sand added to a pure clay increased its erosional strength,
which agrees with existing literature. An addition of microbes to the kaolinite
caused disaggregation of the sediment, which is contrary to some existing
literature. It is clear from this study that further work is required to obtain
a functional relationship between physical properties and erosional strength,
and that cohesive sediments are very sensitive to environmental changes. The
final part of this dissertation presents the results of an experimental investi-
gation of the minimum fluidization conditions for kaolinite clay. A bed of
pure kaolinite was subjected to varying flowrates of water directed vertically
upward through the bed. Few experimental efforts have considered the liquid
fluidization of a purely cohesive material, and the definition of a minimum flu-
idization condition was not clear from a review of literature. Through visual
observation, cracking and separation of the bed into layers was evident, and
in some cases was followed by water ejection through a hole in the bed. The
height of the bed, its volume fraction of solids, and the fluid pressure in the
bed were all measured simultaneously as a function of time. Some correlation
was evident between the pressure drop through the bed and the flowrate of
the water, indicating an asymptotic minimum pressure required to fluidize a
clay bed.
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Chapter 1

General Introduction

1.1 Description of Sedimentation and Fluidization

1.1.1 Sedimentation

Sedimentation is an important process that is encountered in several engineering and scientific disciplines, viz. earth, biological and physical sciences. In coastal and marine environments, which constitute one of the focal points of this research, the sedimentation process is of paramount importance in the formation of the strata layers and transport processes occurring along a margin.

Sedimentation is defined as the coupling of two distinct processes, namely, settling and consolidation of sediment particles. Newly deposited material may be re-suspended due to the water action along the shoreline, and then subsequently deposits due to its own weight during low flow conditions forming a newly consolidating sediment layer. In estuarine environments a significant
amount of re-suspended sediments originates not only from the action of these energetic events, but also from the river deltas and the failure of banks in areas that are prone to erosion (e.g. Wolanski et al., 1988; Evans et al., 1999).

1.1.2 Fluidization

A phenomenon called fluidization has been shown to affect the sedimentation process in marine environments. Fluidization is defined by the upward flow of a fluid (gas or water) through a particulate deposit which supports and entrains particles; it may generate distinctive structures, grain size sorting, and density segregation effects within the fluidized bed.

In marine environments, fluidization occurs in two types, namely unforced and forced. Unforced fluidization occurs during the self-weight consolidation of mud-flats. Forced fluidization primarily occurs due to wave action and during upward gas and liquid propagation; in most cases, gas is produced biogenically under anoxic conditions, and forced liquid propagation is due to hydrologic or geothermal circulation (Schultheiss, 1990).

A ubiquitous feature of aggregative fluidization for both forced and unforced systems is the formation of vertical fluid pipes, known also as “vents”, “fluidization”, or “elutriation” pipes. Fluid vents consist of solid-free zones, which propagate within sediment deposits leaving remnants of “volcano-craters” atop the bed surface as they exit into the water column. The vents are believed to be confined to fairly straight, circular to oval conduits while much of the intervening grain-mass continues in the fixed-bed state. The increased permeability along the vents causes faster dissipation of fluid pressures and faster movement of the fluids relative to the sediment particles.

Some of the impacts of fluidization on near-bed flow processes and bed
properties are believed to be the following:

- Fluidization affects the fluvial erosional strength of sediments by causing particle to particle separation due to the upward fluid propagation. According to Mehta (1989), fluidization itself is a form of erosion.

- The formation of volcano-craters atop a fluidized bed lead to an increase in the bed roughness (Wolanski et al., 1988). Significant volcano-craters typically form during slack conditions right after a high tidal event or during forced gas and liquid propagation.

- A propagating fluidization pipe “dampens” flow turbulence of a current. In this case, the root mean square of the current in the streamwise direction (i.e. turbulence intensity) is decreased approximately by 20 percent (de Wit and Kranenburg, 1997). This impact is applicable to forced fluidization generated by waves.

- The presence of pockets of gas or liquid in the void spaces of sediment retards the self-weight consolidation process of depositing sediments (Wichman et al., 2000). This is in general true for all cases of fluidization that gas or liquid propagation occurs.

Fluidization caused by the self-weight consolidation of depositing sediments or forced fluidization by gas or liquid propagation and the effects of fluidization on the erosional strength of sediments is the focus of this research.

1.2 Environmental Impacts

There are several estuarine and coastal studies around the world that support the argument that fluidization due to upward gas or liquid propaga-
tion has detrimental impacts on the reentrainment of contaminated sediment deposits. Field efforts performed by e.g. the USGS and USACE, to contain contaminated sediment deposits by means of cap materials are questionable since they do not examine the effects of fluidization on the erosional strength of sediments. Existing studies associated to the design of cap materials account only for the shearing action of flow atop the cap layer.

For example, studies in Palos Verde (CA) and in the Hudson River (NY) indicate that the failure of the cap material used in these sites to cover contaminated sediment beds was not caused by the bed shear stress alone but by the fluidization process triggered by water waves and ground water advection respectively. In other areas, such as the Puget Sound (WA) failure of the cap material used to cover oil spills in the old Navy shipyard located in Bremerton, WA was caused not only by the ferry passage but also by the gas propagation within the sediment beds.

A common finding of these studies was that the soil composition of cap layers used to cover the contaminated beds played an important role in promoting the fluidization process. For example, it was found that under the same flow conditions, water content and water chemistry, a bentonite cap layer that has high resistivity to flow action performed poorly. In contrast, a layer comprised by a mixture of sand, bentonite and silt, with relatively lower resistivity to flow than the bentonite layer performed better overall because it allowed the subsurface flow to escape. In the latter case, no pronounced pockets were observed atop the cap layer and retention of the contaminated sediments was attained. The presence of pockets atop the bentonite cap layer introduced a higher level of turbulence around these pockets which led to localized scour of the contaminated bed and eventually failure.

The above discussion on the fluidization process and its effects on sedi-
mentation and sediment stability clearly demonstrates that fluidization should be considered in sediment transport modeling in estuarine environments. Current numerical models (e.g. Syvitski and Morehead, 1999) assume that fluid pressure within the sediment bed is hydrostatic, ignoring the effects of fluidization on sediment strength and sediment properties. Moreover, existing models assume that settling of particles is not affected by the upward motion of the fluid that escapes from the sediment layer. An improved understanding of the fluidization process and the mechanisms triggering this process will aid to the development of physically-based sediment transport models that account for the effects of fluidization on sediment erosional strength.

This research will investigate the onset of fluidization for different types of materials and the effects of fluidization on the erosional strength of those materials.

To isolate the role of different factors affecting the temporal and spatial evolution of fluidization and provide an improved understanding of the mechanisms triggering fluidization, well-controlled laboratory experiments will be performed in a vertical graduated acrylic sedimentation column. The sedimentation column experiments facilitate non-intrusive, unimpeded monitoring of the evolution of fluidized pipes at different temporal and spatial scales (i.e. macro- and microscales) over the depth of a sediment layer. The fluidization column experiments complement other types of fluidization tests, viz. fluidization tests performed in a laboratory flume, where the combined effects of shear action by the flowing water and the fluidization by upward fluid propagation on sediment strength are recorded. Because the emphasis in this research is on fluidization alone and the different processes triggering this process, sedimentation column experiments are deemed to be important to isolate the conditions under which failure of the sediment bed due to forced or unforced fluidization
may occur.

To meet the objectives of this research the laboratory experiments are combined with analytical and numerical work. Analytical work focuses on the possible parameters governing the fluidization process at the microscale level and numerical simulations are performed to simulate the sedimentation process at the macroscale level and provide the average fluid propagation velocities at the onset of fluidization.

1.3 Organization of the Dissertation

This dissertation is organized as follows: Chapter 2 builds on the current literature review considering the conventional approach for treating sedimentation in a vertical column when unforced fluidization is present. Chapters 2 and 4 are interrelated and indirectly address the effects of fluidization on the erosional strength of sediments for forced and unforced fluidization. During testing the role of sediment properties and water chemistry are evaluated. Chapter 3 provides the critical erosional strength values for pure clays and field sediments that represent the onset of bed failure. Chapter 4 offers unique macro and microscale results about the fluidization process, such as description of fluidized pipes, craters, pressure gradients and time series, flow velocities. State-of-the-art equipment and laboratory facilities are employed to provide for the first quantitative information about the onset of fluidization in clay mixtures.
Bibliography


Chapter 2

Experimental and Numerical Observations of the Sedimentation Process of Kaolin: Effects of Fluidization
EXPERIMENTAL AND NUMERICAL OBSERVATIONS OF THE 
SEDIMENTATION PROCESS OF KAOLIN: 
EFFECTS OF FLUIDIZATION

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Authored by:
A. N. Papanicolaou and A. R. Maxwell
2.1 Abstract

The present study considers the sedimentation behavior of a pure clay sediment via laboratory and numerical experiments. Such cohesive sediments are important in environmental applications, as they tend to be a sink for contamination due to their electro-chemical nature. Further, cohesive sediments are typically found in environmentally sensitive ecosystems such as estuaries, whose continued health is critical to the stability of the overall marine and riparian environment. The goal of this study is to determine whether fluidization due to self-weight consolidation of this clay has an impact on overall sedimentation behavior, as the clay transitions from a suspended sediment to a weak soil in the sediment bed.

Various other studies have concluded that the rates of fall and rise of the suspended and deposited sediment interfaces, respectively, are related to the self-weight fluidization phenomena. Through a series of carefully controlled laboratory experiments, the authors show that the variation in these rates are most likely due to mixing techniques or to the presence of entrained gas in the sediment/water mixture. Although self-weight fluidization was observed in most of the tests performed in this study, it did not have a consistent or noticeable effect on the overall sedimentation behavior of the clay.

A numerical simulation using a “black-box” type model was performed in order to examine its suitability in the presence of self-weight fluidization, which is a matter of some question in current literature. The model accounts for the upward movement of fluid in an average sense, and results from the numerical simulation agree well with the results of the experiments, leading to the conclusion that fluidization due to self-weight consolidation does not have a noticeable impact on the rate of descent of the mudline or rate of ascent of
the sediment layer.

The implications are that 1) such numerical models may be used to predict the sedimentation and consolidation behavior under these conditions, if other means of fluidization are not present, and 2) self-weight consolidation does produce fluid flow through the sediment layer, which could lead to further contamination of the water column through dissolution of adsorbed contaminants. The actual amount of fluid flow produced during self-weight fluidization remains to be quantified through further experiments.

2.2 Introduction

2.2.1 Definitions

Gravitational sedimentation is a significant process which has been investigated in several disciplines, including coastal, environmental, chemical, and mineral engineering, hydrology, and geology (Concha and Bürger, 2003). Sedimentation due to gravity is defined as the process whereby a suspension of particles in a fluid settles and consolidates into a sediment layer under its own weight.

Fluidization is a phenomenon that is linked to the sedimentation process (Allen, 1982), and is also known as channelling or fingering flow; it may occur during the collapse of a sediment layer under its own weight, as fluid is squeezed out from the interstices, or may be due to external forcing of some type (as in a fluidized bed). Fluidization may generate distinctive structures, grain size sorting, and density segregation effects within the sediment layer, as observed in various laboratory and field studies (Druitt, 1995; Best, 1989). A ubiquitous feature of such fluidization is a vertical fluid channel, known also as a “vent,”
or “elutriation pipe,” and a hypothetical schematic is presented in Figure 2.1. In some cases, fluid vents consist of solids-free zones which propagate within the sediment layer, leaving remnant volcano-like structures atop the sediment layer as they exit into the overlying fluid. Such pipes may also take a sheet-like form, extending through the solids layer in all directions (Roche et al., 2001).

In marine and coastal environments, fluidization can be initiated by a variety of mechanisms, including wave activity, seismic activity, upward gas propagation, and liquid propagation due to geothermal circulation or self-weight consolidation (e.g. Dionne, 1973; Yamamoto et al., 1978; Schultheiss, 1990; Yun et al., 1999). The primary application considered in this study is the self-weight fluidization of underconsolidated estuarine muds, caused by the self-weight consolidation of newly deposited sediment as depicted in Figure 2.2. This figure shows the process of sediment deposition and re-entrainment, coupled with the self-weight fluidization process. Fluidization pipes are believed to form when the fluid which is trapped within the sediment structure is expelled as the pore fluid pressure increases during consolidation, and the sediment structure rearranges itself to accommodate the increasing external load of its

Figure 2.1: Schematic of volcanic fluidization with vertical elutriation pipes, after Mount (1993).
own weight. Particularly in estuaries and deltas, with a constant influx of predominantly soft muds (Kuehl et al., 1988; Allison et al., 1995), self-weight fluidization of sediment is an ongoing process. Further, it is believed that channels (pipes) produced by self-weight fluidization may act to generate fluidization at a larger scale, which may be caused by the external mechanisms mentioned previously (Roche et al., 2001). Fluidization occurring at a larger scale in turn affects the erosional strength of deposited sediment by rearranging the sediment structure (Mehta, 1989). Some researchers have also indicated that fluidization pipes which elutriate into a water column may lead to an increased settling rate of suspended aggregate particles (Vesilind and Jones, 1990), and may also affect near-bed turbulent flow characteristics by changing the roughness of the sediment bed through the formation of craters (de Wit and Kranenburg, 1997).

The long-term objective of the research presented here is to provide a
functional relationship between fluidization characteristics and physical properties of the fluidized mud such as water content, sediment composition, and clay type. Such a functional relationship will facilitate a description of the behavior, and is very much needed for modeling nearshore sediment transport and fate of contaminated sediments (e.g. Lee and Mehta, 1997). In order to achieve this long-term objective, reliable procedures must be developed in advance, in order to facilitate adequate detection of self-weight fluidization within a sediment layer.

2.2.2 Critical literature review

Up to the present day, the characteristics of the self-weight fluidization process have primarily been investigated in the laboratory by performing batch sedimentation tests.

In general, researchers have pointed to the mudline interface variation with time (batch curve) and solids flux vs. volume fraction of solids (flux curve) as indicators of the occurrence self-weight fluidization (e.g. Vesilind and Jones, 1990; Fitch, 1993). According to Been (1980); Vesilind and Jones (1993); Fitch (1993); Holdich and Butt (1995b), during batch sedimentation tests of certain slurries, short, upward-moving channels were observed. With the meeting of the ascending zone of channels and the descending mudline interface is a concomitant observation of an increase in the interfacial (mudline) settling velocity and therefore an increase in the flux of the settling material (Vesilind and Jones, 1993). A conceptual sketch illustrating the effects of self-weight fluidization as described by (Vesilind and Jones, 1993) is shown in Figure 2.3. Case 1 in Figure 2.3 refers to a sedimentation test in the absence of self-weight fluidization, and depicts typical mudline interface vs. time,
sediment-suspension interface vs. time (L-curve), and velocity vs. time curves. Case 2 in Figure 2.3 refers to a sedimentation test with self-weight fluidization. According to Vesilind and Jones (1990), when fluidization is present, a pronounced inflection point is formed in the descending mudline interface between the first and second falling rate regimes, and the flux curve obtains a doubly-concave shape (C–D), indicating an increase in the rate of settling of
material relative to Test 1. Other researchers (e.g. Gaudin et al., 1959) agree with the fact that volcanoes form at the mudline interface within the first and second falling rate region (C–D); however, they noticed no inflection point at the mudline interface despite recording the eruption of small flocs.

Recently, Maxwell et al. (2003) performed a series of preliminary batch settling tests for a kaolinite sediment mud and found that during self-weight fluidization, no inflection point was recorded in the mudline interface curve. Maxwell et al. (2003) noted that formation of fluidization occurred in the early stages of a test, and not within the second falling rate region as other researchers have reported. Volcanoes, however, were not visible until the supernate had cleared; this generally took place after the second falling rate region. Lastly, Holdich and Butt (1995b) noted that the top of the fluidized region coincided with a solid concentration characteristic and indicated that stirring had an effect on channel formation and ultimately on the settling rate of suspensions of calcite. Holdich and Butt (1995b) observed an inflection point in the mudline interface curve only for the stirred test.

The above literature review suggests that while the majority of research completed in the area of self-weight fluidization agrees with the notion that batch sedimentation tests may be the best approach in examining fluidization in a controlled environment, the results are inconsistent across various studies. There are several reasons for these inconsistencies. One of the difficulties inherent in sedimentation tests is developing a mixing technique which allows repeatable results. Cole (1968) found that compressed air was unreliable as a mixing technique, and that using a pump to recirculate the mixture through a tank was more effective. Jones (1986) used a similar recirculating method for a calcium carbonate slurry, while Michaels and Bolger (1962) used 8–10 repeated inversions of a test cylinder for their kaolinite slurry or mixed it in
a Waring Blender for one minute. Fitch (1993) noted self-weight fluidization (channelling) in sedimentation tests and attributed changes in the batch and flux curves to the presence of channels; however, he used a glass rod to stir the sediment, or mixed it by repeated inversions of the sediment column, both of which are relatively gentle means of stirring. Recently, Channell et al. (2000) indicated that the time to formation of a structure in a sedimenting suspension is strongly dependent on the stress history of the suspension as well as its chemical composition. The implication for mixing methods is that a high-speed mixing process may disrupt the structure and remove the dependence of the gel point of a suspension on its initial solids concentration, which would improve repeatability. Consequently, it is possible that the variations in batch and flux curves observed in prior studies are primarily due to unreliability of mixing techniques, rather than fluidization per se. Other questions that need to be addressed are the efficacy of conventional tools to describe the onset of self-weight fluidization and the role of the sediment type and initial conditions (concentration, slurry height, chemistry) in affecting the temporal and spatial characteristics of fluidization.

Because one of the intents of performing the batch sedimentation tests is to study fluidization under controlled conditions, the effects of mixing on self-weight fluidization need to be re-examined. Furthermore, it is important to ensure that certain mixing approaches do not introduce other types of fluidization that could mask the effects of self-weight fluidization (e.g. certain types of mixing can entrain air, which may in turn lead to bubble propagation). This will defeat the purpose of running batch tests for self-weight fluidization identification and description.
2.3 Objectives and Methodology

The overarching objective of this research is to develop a blueprint methodology for batch sedimentation tests that allows qualitative and quantitative identification of self-weight fluidization by removing the effects of mixing on the evolution of fluidization. Secondary objectives of this investigation include: (1) examination of the efficacy of conventional tools to describe fluidization and (2) investigation of the role of initial material properties and height on fluidization.

To meet the objectives of this investigation, two methods of mixing were used, namely, pneumatic mixing by compressed air and rotary mixing by high-speed propeller to elucidate the role of mixing on the formation of channels. The utility of vacuum techniques was examined for minimizing the effects of air-bubble propagation on liquid channels.

Qualitative preliminary sedimentation tests were performed at initial concentrations of 10, 25, 50, and 75 g/L using visual observations, in order to gain familiarity with the procedure and approximate the time of occurrence of self-weight fluidization. Two cases, 75 and 125 g/L (which represent the conditions found in an estuarine environment), were examined in greater detail with the aid of advanced instrumentation in order to address the objectives of the study as outlined earlier.

A comparison of these two cases will elucidate the role of initial density on the sedimentation and fluidization processes. Case 75g/L was performed for different initial heights to isolate the role of height on these processes. All comparisons were made for the aforementioned mixing techniques.

The advances made in imaging technology in recent years allowed use of this technology for studying sedimentation processes (e.g. Zhu et al., 2000).
Quantitative measurements of the visible features such as the upward-propagating fluid velocity within the channels was carried out for tests 75 and 125 g/L with NIH ImageJ, a Java-based image processor available from http://rsb.info.nih.gov/ij/. In addition, visual measurements (naked eye, camera) of the variation of the mudline interface with time were complemented with an automated gamma radiation scanning system. The gamma system results minimize the subjective error which is inherent in visual observation of the mudline interface and the region where inflection points in the mudline interface may occur. The efficacy of more “sensitive charting tools” were considered for tests 75 and 125 g/L, to identify when fluidization occurs.

To explore the efficacy of alternative techniques for identifying fluidization, the experimental component of the research was complemented with numerical modeling. An existing batch sedimentation model developed by Diplas and Papanicolaou (1997) and later enhanced by Papanicolaou and Diplas (1999) was employed in order to (1) simulate the sedimentation process for tests 75 and 125 g/L by reproducing the mudline interface and sediment-suspension interface over time, and (2) determine the minimum required fluid velocity to initiate self-weight fluidization for the two kaolinite tests. While a detailed description of the 1-D sedimentation model is included in Diplas and Papanicolaou (1997) or Papanicolaou and Diplas (1999), certain aspects of the model (e.g. assumptions and hypothesis) which are germane to the present case are provided below. The model equations presented below were deduced based on the following considerations:

1. The presence of a granular medium with elastic properties, described by the constitutive equations of Tiller and Khatib (1984).

2. The settling and consolidation processes occur at the particle assembly
level (macroscale level) rather than the particle level (microscale level).

3. Fluidization commences when mechanical parting of particles is present, *viz.* the effective or normal contact stress is equal to zero (e.g. Jackson, 2000)). In the model at each stage of fluidization or defluidization, the particle assembly was envisaged to be at rest, in the sense that the local average velocity of particles vanishes everywhere.

The governing equation of the self-weight consolidation process, Equation 2.1, is a nonlinear, transient partial differential equation of the parabolic type, derived in the Eulerian coordinate system:

\[
\frac{\partial \sigma'}{\partial t} - \frac{K_o (1 + a\sigma')^{(1-\delta)}}{a\mu\beta} \left( \frac{\partial^2 \sigma'}{\partial z^2} \right) - \frac{g\Delta\rho\varepsilon_{so} K_o (2\beta - \delta)(1 + 2\sigma')(\beta - \delta)}{\mu\beta} \left( \frac{\partial \sigma'}{\partial z} \right) - \frac{K_o (\beta - \delta)(1 + a\sigma')^{-\delta}}{\mu\beta} \left( \frac{\partial \sigma'}{\partial z} \right)^2 = 0 \quad (2.1)
\]

In Equation 2.1, \( \sigma' \) is effective stress, \( \varepsilon_{so} \) is the null stress volume fraction of solids, \( K_o \) is the intrinsic permeability of the sediment layer, \( a, \beta, \) and \( \delta \) are compressibility coefficients, \( z \) is the Eulerian coordinate distance from the cylinder bottom, \( \mu \) is dynamic viscosity, and \( \Delta\rho \) is \( \rho_s - \rho \), where \( \rho_s \) is solids density. In order to predict the variation of the sediment-suspension and mudline interfaces, Equation 2.1 was solved using the finite element and finite difference methods to provide the distribution of \( \sigma' \) within the sediment layer (Papanicolaou and Diplas, 1999). In the spatial discretization of the domain, the Petrov-Galerkin method was used to obtain the weak form of Equation 2.1. The Crank-Nicolson scheme was employed to perform the model’s time discretization, and the contribution of the non-linear terms was determined by employing the standard Newton-Raphson iteration scheme. The
following boundary conditions were used: At the sediment-suspension interface (L-curve), \( \sigma' = 0 \). At the bottom of the sediment layer, the natural boundary condition describes the impermeable base of the sediment column \((z = 0)\), where the gradient of effective stress is

\[
\frac{\partial \sigma'}{\partial z} = -g (\rho_s - \rho) \epsilon_{so} \left( 1 + \frac{\sigma'}{\sigma_o} \right)^\beta \tag{2.2}
\]

The initial condition of effective stress at the beginning of a time step is given by \( \sigma' = f(z) \), where \( f(z) \) is a known function. Once the distribution of \( \sigma' \) is obtained by Equation 2.1, the height decrease \( \Delta l \) of a layer with initial height \( l_k \) due to sediment consolidation is given by Equation 2.3

\[
\Delta l_k = l_k \left( \frac{a \beta}{1 + a \sigma'_{sk}} \right) \sigma'_{sk} \tag{2.3}
\]

where \( a \) is a compressibility coefficient and \( \beta \) is as defined previously. The total decrease in the height of the sediment layer that consists of \( n \) sublayers over time is defined by Equation 2.4 as

\[
\Delta L = \sum_{k=1}^{n} \Delta l_k \tag{2.4}
\]

The height of the sediment-suspension interface at the end of a period \( \Delta t \) is determined by

\[
L = L_t + v \Delta t \tag{2.5}
\]

where \( v = \Delta L / \Delta t \) denotes the velocity of a characteristic line emanating from the L-curve. According to Font (1991), these lines are tangent to the L-curve. The height of the mudline interface is equal to

\[
H = L + v \Delta T \tag{2.6}
\]

where

\[
\Delta T = \frac{\phi_{so} H_o - \int \epsilon_s dz}{\phi_s (v - u_{so})} \tag{2.7}
\]
where $\phi_s$ is the suspended sediment volume fraction of solids, $\epsilon_s$ is the sediment layer volume fraction of solids, and $u_{so}$ is the initial settling velocity of the suspension. The results deduced by the numerical model, the mudline interface determined via Equation 2.6 were compared against the experimental data.

In order to determine the minimum fluid velocity for the initiation of self-weight fluidization Papanicolaou and Diplas (1999) combined the balance of static forces on a control volume, the modified Darcy’s law, and the mass balance equation for the fluid and solid phase assuming negligible friction between the particles and the cylinder wall. They deduced the following equation, which is applicable to a one-dimensional consolidating bed,

$$u_f = \frac{K \epsilon_s}{\mu (1 - \epsilon_s)} \left( -\frac{\partial \sigma'}{\partial x} - \gamma_f (1 - \epsilon_s) + \gamma_s \epsilon_s + \gamma_f \right)$$

(2.8)

where $\gamma_f$ is the specific weight of the fluid, $\gamma_s$ is the specific weight of the solids, and other variables have previously been defined.

### 2.4 Experimental Setup

#### 2.4.1 Material used

The sediment used in this study was prepared from dry kaolin powder (Hydrite PX, Georgia Kaolin Company) with typical median particle size of 0.68 $\mu$m and specific gravity of 2.58, according to the manufacturer. A specific gravity test placed the actual value at 2.63, and a value of 2.6 was used in calculations. Kaolinite is a 1:1 clay mineral consisting of layered tetrahedral (typically silica) and octahedral (typically alumina) sheets, bonded by hydrogen bonding between hydroxyl and oxygen ions of adjacent layers, as well as van der Waals attraction. Due to these bonds, kaolinite is a stable clay
mineral (van Olphen, 1977), with low cation exchange capacity (CEC) and surface area (McBride, 1994). As kaolinite layers tend to not separate except in “extremely polar solvents,” (McBride, 1994), it should give well-defined, repeatable results for settling and consolidation. Kaolinite has also been widely used in laboratory studies (e.g. Michaels and Bolger, 1962; Cole, 1968; Dell and Kaynar, 1968; McConnachie, 1974; Austin and Challis, 1998), and thus a range of literature for qualitative comparison exists. Tap water of near neutral pH (7.6) was used in this study.

2.4.2 Facilities

The sedimentation tests were conducted in the Albrook Hydraulics Laboratory of Washington State University. Sedimentation columns were constructed of cast acrylic tubing, 140 mm ID by 152 mm OD, and adhesive rules graduated in millimeters were placed on each column. According to Cole (1968), diameter (wall) effects are minimal for this size and larger diameter columns. The tallest column was 2 m and the shortest was 0.25 m in height, giving a wide range of possibilities.

Sedimentation scanning

Gamma radiation was used in the present study to measure the volume fraction of solids over time, following a method similar to that of Been (1982), who presents a very detailed description of the construction and testing of an X-ray system for this purpose. The gamma source offers advantages over other techniques including sampling taps on column walls as used by Vesilind and Jones (1993) and electrical conductivity measurements as used by Holdich and Butt (1995a) in that it facilitates non-destructive spatial and temporal
measurements without affecting sediment microstructure. The fundamental principle behind this technique is that increasing amounts of solids absorb more photons. For a narrow beam of monochromatic radiation, the attenuation of photons is described by Beer’s Law, given in Equation 2.9:

\[ I = I_0 e^{-(\eta/\rho_m)x} \]  

(2.9)

where \( I \) is the intensity of radiation (counts per second) after attenuation, \( I_0 \) is the intensity before attenuation (source intensity), \( \eta \) is an attenuation coefficient, \( \rho_s \) is the density of the material, and \( x \) is the distance between the source and detector. The implication is that a constant intensity radiation source, when combined with a constant material thickness, can be used to determine the attenuation coefficient of a given material; for a monochromatic source, this attenuation coefficient is constant, which allows density to be measured directly by calibrating the system appropriately. For the present study, a 550 mCi \(^{241}\)Am gamma source (60 keV) was used to provide the radiation beam, and a Harshaw 6S2/2-X NaI(Tl) detector with integrated photomultiplier was used for detection of the radiation. The signal from the detector was amplified and then passed through a single-channel analyzer (Harshaw NC-22) operated in windowed mode to filter out noise. A Harshaw NS-30 scaler was used as a counter, and was controlled manually for calibration or automatically by a computer program.

Collimation is a key element of radiation scanning (Been, 1982), and in this case was provided by a lead plate 9.5 mm thick with a 6.35 mm circular hole for the source beam, and a 0.889 by 36.8 mm slit for the detector, machined in a 31.8 mm deep block of lead. These are shown on opposite sides of a sedimentation column in Figure 2.4. Vertical motion of the source and detector was provided by a step motor, and measurements were taken at discrete points.
A QuickBASIC program controlled the stepper motor and gamma counter; the photon count was recorded at each specified height, over a two-second interval (the interval was determined via a $\chi^2$ test according to Knoll (1979)) beginning at a specified time. The time between traversals was set at a time interval of $\sim$10–20 minutes, depending on the total traverse time, and a given test could last up to three weeks. By examining profiles and comparing with visual observation, the spatial accuracy of the system was ±1 mm at a 5 mm scan interval, with respect to determining the location of the mudline interface. The system was calibrated before each experiment by mixing suspensions of known solids content and recording the gamma count rate; these data were then fit to an equation of the form

$$\phi_s = K_1 e^{-K_2 I} - K_3$$

(2.10)

where $\phi_s$ is the volume fraction of solids and $K_n$ are constants which describe the attenuation characteristics of the material. Error of 3–5% of volume fraction of solids was typical, and was highest at very low volume fractions, due to the statistical nature of radiation interactions. A Sony DFW-X700 digital camera equipped with an optical zoom macro lens was used to capture images of the sedimentation process, in order to complement the gamma system.
readings with respect to the mudline height variation with time. This camera outputs square-pixel images at 1024x768 resolution, directly to a computer hard drive via an IEEE-1394 (FireWire) interface. Fluidization activity was captured at the highest possible frame rate (15 frames per second), while overall mudline settling was captured at much longer intervals (10 minutes to 1 hour, depending on the region of the mudline and concentration of the test).

The camera was controlled by intervalometer software which captured each image with a time stamp at specified intervals. The images were renamed, sorted, and numbered sequentially with a custom computer program written for this purpose, in order to enable import of the images into QuickTime (http://quicktime.apple.com/) to view the image sequence as a continuous movie. Adjustment of image histograms was performed using TIFFany3 on Mac OS X, which allows flexible batch processing of images. Frame sequences were analyzed using the FlowJ plugin for ImageJ developed by Abràmoff et al. (2000).

**Mixing**

The authors of the present study sought to elucidate this problem by using two means of mixing, in hope of providing some resolution to the inconsistencies and repeatability issues observed in current literature. In order to do this, two methods of mixing were used: compressed air and a high-speed propeller. Compressed air was utilized by connecting an air hose to section of perforated PVC pipe, which was then used to vigorously stir and bubble the kaolin-water suspension directly in the settling column. Unfortunately, it was very difficult to produce repeatable results with this pneumatic mixing method. A second mixer was constructed for the present study (Figure 2.4)
using a 1725 RPM motor to drive a 95 mm propeller; due to the size and speed of this system, it was necessary to mix the kaolin-water suspension in a separate container, in order to avoid breaking the test cylinder. A small rotary vane pump was then used to transfer the suspension from the mixer to the settling column, a process which took only a few seconds to complete. In order to determine if weathering of the kaolinite particles occurred during the mixing process, particle size analyses were performed (using a Mastersizer, Malvern Instruments Ltd., Worcestershire UK) on samples mixed for 5, 10, 15, and 20 minutes; no significant difference in particle size was observed by varying the mixing duration. Finally, a single test with initial concentration of 125 g/L was mixed using a Jiffy wand mixer (Jiffy Mixer Co., Inc., Riverside, CA), which is a rotary mixer designed for effective mixing with low air-entrainment. The Jiffy mixer was mounted on an electric drill, and gave a visually-uniform appearance to the slurry, while it appeared to entrain less air than the propeller mixer.

2.4.3 Experimental Procedure

Prior to testing with the gamma system, it was necessary to calibrate the equipment with known concentrations of kaolinite in water. For this purpose, mixtures were prepared at 0, 25, 50, 100, 150, 200, and 400 g/L, in an acrylic cylinder of identical diameter and wall dimensions as the test cylinder. This cylinder was placed in the appropriate location in the traversing system, and three replicate gamma counts of 2 s. duration were taken for each mixture. An exponential curve fit to concentration vs. count rate resulted in an equation similar in form to Equation 2.10, which could be used for post-processing the raw count data from the gamma system. After calibrating the gamma
equipment, the settling column was carefully cleaned, and steps were taken to ensure that all equipment was ready to start as soon as the clay-water suspension was prepared. For a specified initial concentration, the appropriate volume of water was measured, using a settling column as a graduated cylinder. Likewise, the appropriate mass of kaolin powder was weighed, and placed in the mixer.

In each of these tests, dry kaolin powder was mixed with tap water for a minimum of five minutes, either by compressed air or a rotary device. Samples mixed by means of compressed air were mixed in the sedimentation column, while samples mixed with the rotary mixer were mixed in an external vessel for 5 minutes and then pumped into the sedimentation column already in place for measurements (Figure 2.4). Observations of the mudline interface were made visually (by naked eye or monitoring with the camera) and indirectly with the gamma profiling system. Vacuum was not applied to any of the 75 g/L tests for degassing. It was not feasible to distinguish the L-curve by visual means and the gamma profiling system was used for this purpose.

2.5 Results

2.5.1 Experimental

Table 2.1 summarizes only the results of the experiments which were performed at initial concentrations of 75 and 125 g/L. For the sake of brevity, the remaining tests performed in the course of this study (at initial concentrations of 10, 25, and 50 g/L) are not presented; however, it must be noted that the results of the 75 g/L tests are typical of the results of the other tests performed under similar conditions.
<table>
<thead>
<tr>
<th>$\phi_{so}$</th>
<th>$C_o$ (g/L)</th>
<th>$H_o$ (mm)</th>
<th>$\bar{u}_{so}$ (mm/s)</th>
<th>$T_p$ (sec.)</th>
<th>Fluidization</th>
<th>Mixing</th>
<th>Measure</th>
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<td>250</td>
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<td>Vis</td>
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<td>250</td>
<td>0.024</td>
<td>5000</td>
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<td>Rotary</td>
<td>Vis/\gamma</td>
</tr>
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<td>Vis</td>
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<td>500</td>
<td>0.018</td>
<td>16000</td>
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<td>Vis/\gamma</td>
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<td>Vis</td>
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<td>16500</td>
<td>Yes</td>
<td>Rotary</td>
<td>$\gamma$</td>
</tr>
</tbody>
</table>

Table 2.1: Summary of experimental results for the tests discussed in this article. NR indicates no visual record of fluidization.

In Table 2.1, “$C_o$” refers to initial concentration, “$H_o$” refers to initial height, “$\bar{u}_{so}$” is the average initial settling velocity determined from the gradient of the linear increment of the mudline interface, “$T_p$” is the time spent in the initial (linear) rate period, “Fluidization” denotes the presence or absence of self-weight fluidization based on visual/camera observations, “Mixing” denotes the type of mixing, and “Measure” indicates whether purely visual measurement or the gamma system was used.

Several points are immediately obvious upon examination of Table 2.1. First, the solids settling velocities of the tests with greater initial height (1000 and 1500 mm) show greater variation in settling velocity magnitude with mixing technique than those of lesser height. Second, settling velocities were higher for the rotary-mixed tests than for the pneumatically mixed tests (with the exception of the $H_o = 250$ mm tests). This reduced mudline velocity in the pneumatic mixing cases is possibly due to buoyant effects of entrained air, or to a reduced effective settling area due to bubble coalescence. Repeatability of the pneumatically-mixed cases was virtually impossible, particularly as ini-
tial height and concentration increased; this is attributed to the difficulty of uniformly mixing the sediment and water over the entire height of the column with the pneumatic mixer, as well as the effects of sediment stress history which can be removed by high-shear mixing methods (Channell et al., 2000).

For the rotary mixed tests it was found that both visual (camera) and gamma measurements of the mudline interface were identical. This was confirmed by repeating the rotary mixed tests and plotting the batch curves in the same figure for comparison purposes. Figures 2.5 and 2.9 demonstrate the close agreement of the visual and gamma measurements for tests 75 g/L and 125 g/L.

![Graph](image)

Figure 2.5: Comparison of visual observation and gamma system measurement of mudline interface for the 75 g/L rotary-mixed test.

**75 g/L initial concentration results**

Figure 2.6 shows the results of all of the tests conducted with an initial concentration of 75 g/L. The pneumatic and rotary mixed tests have the same
Figure 2.6: Mudline and sediment curves for sedimentation tests of kaolinite at initial concentration of 75 g/L
final sediment height, as expected based on the conservation of mass. The initial settling rates in the 250 and 500 mm initial height tests are also the same, but the taller tests (1000 and 1500 mm initial heights) exhibited very different initial settling rates; the pneumatically mixed tests settled slower, for the reasons previously postulated. As self-weight fluidization primarily occurs in the early stages of batch sedimentation (e.g. Mishler, 1918; Been, 1980; Vesilind and Jones, 1993; Holdich and Butt, 1995b), it is likely that if self-fluidization has an effect on sedimentation, it will itself be significantly affected by the mixing type and initial test conditions. It was not possible in the present study to separate the effects of self-weight fluidization from those of mixing or initial conditions, and it is questionable whether this is possible in any case.

Glasrud et al. (1993) investigated the effects of degassing suspensions of iron oxide settling in mineral oil, and found that suspensions with gas have an initial settling rate that is directly proportional to the initial volume fraction of solids, i.e. \( u_{so} \propto \phi_{so} \). On the other hand, they found that the settling velocity of degassed suspensions was inversely proportional to the initial volume fraction of solids, i.e. \( u_{so} \propto 1/\phi_{so} \). Further, Glasrud et al. reported that degassed suspensions of iron oxide were more subject to variation between test runs; their conclusion was that gas bubbles help to “regularize the collapse” of the sedimenting particles, somehow. The findings of the present study support the general belief that gas entrainment has an effect on repeatability of experiments, but do not support the specific findings of Glasrud et al.. It is not clear whether pneumatic mixing necessarily entrains more air than the high-speed rotary mixing process, although this appeared to be the case in the present experiments. If pneumatic mixing does entrain more air, as the authors believe, then the indication is that material type (solid and fluid) determines
the behavior of the suspension in the presence of entrained gas; in the case of Glasrud et al., their fluid phase was a mineral oil, which had a higher viscosity than the water used in the present study. The increase in velocity with solids fraction reported by Glasrud et al. (1993) in mineral oil produced results which are contrary to most experimental findings, which indicate that settling velocity increases with decreasing volume fraction (e.g. Cole, 1968; Kynch, 1952; Vesilind and Jones, 1990, 1993; Holdich and Butt, 1995b,a). The formulation of Richardson and Zaki (1954) (Equation 2.11)

\[ u_s = u_\infty (1 - j \phi_o)^n, \quad n > 1 \]  

(2.11)

where \( j \) is the aggregate volume index (ratio of total aggregate volume to solids volume) and other variables have previously been defined, also indicates clearly that sedimentation velocity decreases with increasing volume fraction of solids. Possibly the viscosity or surface tension effects dominated in the case of Glasrud et al. (1993), and the shape of the iron oxide particles may also have been a contributing factor. Further, the effect of mixing is unclear; low-shear mixing methods, as discussed previously, can produce unreliable results, and Glasrud et al. only note that their suspension was “well-stirred.”

The effects of self-weight fluidization are not consistent with regard to the batch curves in the present study. In general, current literature supposes that fluidization channels increase settling velocity, leading to slope changes in the mudline (e.g. Vesilind and Jones, 1990; Holdich and Butt, 1995a), but this is not evident in the present case. In order to provide a more sensitive comparison, some authors have used a flux plot (\( \phi_s u_s \) vs. \( \phi_s \)) or a plot of \( u_s \) vs. \( t \) (e.g. Gaudin et al., 1959; Tory, 1961; Holdich and Butt, 1995a; Fitch, 1993). A plot of \( u_s \) vs. \( t \) is presented in Figure 2.7, where the mudline and its derivative with respect to time are plotted for both the pneumatic-visual
and rotary-gamma versions of the 75 g/L-1500 mm test. The derivative was computed by interpolating the mudline to produce evenly spaced points, then taking the derivative using a central difference scheme in Igor Pro (http://www.wavemetrics.com/). The standard batch height is given in mm on the right-hand side, while the interface velocity is given in mm/s on the left-hand side; they are plotted as functions of time, each corresponding to the other. The rotary-gamma test exhibits higher average velocity, on average.

![Graphs of interface velocities](image)

Figure 2.7: Comparison of interface velocities of two tests at initial concentration of 75 g/L, computed by taking the derivative of the mudline with respect to time.

The fluctuation in the velocity vs. time curve is an artifact of plotting the mudline by interpolating the density profiles from the gamma system over the duration of the test. The resulting density contours are not a perfectly smooth curve. Self-weight fluidization was observed in both cases, but did not produce a significant change in the overall trend of the interface velocity plot, contrary to the observations of Vesilind and Jones (1990) and Holdich and Butt (1995a). Perhaps this is attributable to the fact that Vesilind and
Jones (1990) and Holdich and Butt (1995a) both used calcite slurries, rather than the kaolinite used in the present study. Other tests (not shown here) conducted by the authors showed local maxima in the interface velocity curve which did not correspond to self-weight fluidization. Hence, the batch curve and flux curve are not a reliable method for the present purpose of detecting self-weight fluidization.

**Image Analysis Results**

A photograph taken during sedimentation of the 75 g/L rotary mixed test is shown in Figure 2.8(a). Maximum vertical velocities at the wall appear to be \(\sim 3 \text{ mm/s}\), as seen in the darkest regions. FlowJ uses in indexed RGB color wheel to show the magnitude and direction of flow, shown as inset in Figure 2.8(b). In these images, dark regions indicate vertical upward flow, while light regions indicate vertical downward motion; the vectors drawn on the images indicate flow direction, irrespective of flow magnitude.
125 g/L initial concentration results

In the case of the 125 g/L-500 mm test performed using the gamma system for density profiling, the air was removed using the vacuum method described earlier, which reduced the amount of entrapped air by a visible amount. Figure 2.9 shows the isoconcentration lines detected by the gamma system, plotted with the visually observed mudline from the second test. The isoconcentration lines were plotted using Igor Pro, by interpolating between density values in the height-time plane. Height is plotted on the vertical axis, as a function of time (horizontal axis), and the contour parameter is concentration in grams per liter. Significant bubble attachment to the walls of the cylinder was evident in the visual-only test, which would lead one to suspect that settling rates would be decreased, based on the results observed in the 75 g/L initial concentration tests. Inspection of the settling rate data in Ta-
ble 2.1 seems to contradict this, however. Two possible reasons are offered: the effects of vacuum cannot be compared directly with the effects of pneumatic mixing, although both involve gas removal or addition; also, mixing methods were slightly different, as a new low-air entrainment mixer was used in the visual-only 125 g/L-500 mm test. Self-weight fluidization was likewise observed in both of these tests, once again with no systematic local variations in batch curves which could be attributed to fluidization. A photograph of a remnant volcano, taken when the mudline interface was at 210 mm is shown in Figure 2.10, and is a clear example of the remnant volcanoes from the self-weight fluidization process; at the time that this photo was taken, fluid motion was not evident. Some remnants of air bubbles (pockets) were also visible on the mud surface.

![Volcano](image.png)

Figure 2.10: Photograph of a remnant volcano, in pure kaolinite. Initial concentration of 125 g/L, initial height of 500 mm; scale markings visible in lower-left corner are 1 mm apart.

2.5.2 Numerical Results

Knowing that self-weight fluidization was present in many of these tests (see Table 2.1), it was desired to compare the results of the experiments where fluidization was pronounced with a numerical simulation. The authors are unaware of a model which explicitly accounts for self-weight fluidization at
this time. For the present study, the model developed by Diplas and Papanicolaou (1997) and further described by Papanicolaou and Diplas (1999) was used. The model considers 1-D sedimentation, which accounts for the dynamic interaction of the settling of solids in the suspension region and self-weight consolidation. The settling process of solids is modeled by the theory of Kynch (1952), and the consolidation by a self-weight non-linear finite strain model. The coupling of the two components is accomplished by continuously accounting for the effect of the newly deposited sediment on the thickness of the sediment layer and the release of upward moving fluid and its influence on the settling velocity of solids within the suspension. Some of the methodological advances of this model in comparison with others (e.g. Tiller, 1981; Den Haan, 1992) is that (1) it accounts for the release of the upward moving fluid in an average sense and (2) it is applicable to low stress conditions (<10 MPa). Current sedimentation models (e.g. Liu and Znidarčić, 1991) typically underpredict the time required for the compaction of sediments and do not provide accurate predictions of the density and pore pressure distributions when infinitesimal strains occur.

Two of the experimental cases were chosen for numerical simulation: the 125 g/L-500 mm test and the 75 g/L-1500 mm test. The parameters used in the model are shown in Table 2.2.

The results of the simulation of the first case are shown in Figure 2.11. This figure shows the experimental results from the gamma test, plotted with the results from the numerical test. In this case, the gamma test was rotary-mixed and vacuum was applied before sedimentation. The model predicts the suspension and consolidation very well, using the parameters shown in Table 2.2.

The 75 g/L-1500 mm test results are shown in Figure 2.12. The numer-
Table 2.2: Parameters used in numerical simulation cases

<table>
<thead>
<tr>
<th>Parameter</th>
<th>125 g/L-500 mm</th>
<th>75 g/L-1500 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>$1/\alpha$</td>
<td>150</td>
<td>200</td>
</tr>
<tr>
<td>$\epsilon_{so}$</td>
<td>0.07</td>
<td>0.062</td>
</tr>
<tr>
<td>$\beta$</td>
<td>0.323</td>
<td>0.323</td>
</tr>
<tr>
<td>$\delta$</td>
<td>1.145</td>
<td>1.145</td>
</tr>
<tr>
<td>$K_o$</td>
<td>$6 \times 10^{-13}$</td>
<td>$8.5 \times 10^{-13}$</td>
</tr>
<tr>
<td>$\rho_s$</td>
<td>2630</td>
<td>2630</td>
</tr>
<tr>
<td>$\mu$</td>
<td>0.001</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Figure 2.11: Numerical simulation compared with experimental results for the 125 g/L-500 mm case
2.5.3 Fluidization

The effects of self-weight fluidization are not directly considered in the numerical model, although rising fluid flow is generally accounted for. Knowledge of the exact duration and rate of fluid flux through the porous medium would be necessary to precisely model self-weight fluidization, and this information is not presently available. As a “black-box” model, the present simulations seem to be unaffected by the presence of strong fluidization which was present in these tests (see Figure 2.10). In an overall sense, the present model may be able to quantify the sedimentation behavior of a fluid mud, if the necessary
boundary conditions are known. However, the onset of self-weight fluidization cannot be predicted, and it is possible that extreme events of fluidization (by external means such as wave action, ship motion, or subsurface flow) will cause significant changes in the overall behavior of the sedimenting mud. This needs to be examined further.

The results of the numerical model experiments further validate the observation of the authors that for the materials in question (kaolinite and water), self-weight fluidization cannot be detected by “traditional” means of observing the batch sedimentation curve or even the more sensitive flux curve, as the model’s bulk property predictions agree with the experimentally measured properties. Hence, a more sophisticated examination of microstructure and small-scale processes will be necessary to determine the effects of fluidization.

2.6 Conclusions

The present study examined the results of laboratory experiments in batch sedimentation of kaolinite clay, at concentrations typical of estuarine and nearshore sediment environments, which are often contaminated or at high risk for contamination. The aim was to examine the impact of self-weight fluidization (channelling) on sedimentation characteristics of clay under these conditions, and particular emphasis was placed on repeatability of experiments and correlation with past work. It was found that mixing and entrained gas can have significant effects on the sedimentation behavior of otherwise similar mixtures, but the effects are somewhat unpredictable. In general, mixing with a high-shear device such as a propeller produced repeatable experimental conditions, whereas mixing by compressed air did not. The effects of entrained gas have not been fully determined, but it appears to retard the settling pro-
cess; in the cases of shorter initial height (250 and 500 mm), entrained gas also reduced the final sediment height, which implies a more compacted sediment. In addressing these experimental issues, the authors hope to generate some discussion of the appropriate means of conducting such tests, as it appears difficult to reproduce extant tests. It is possible that some authors have mis-attributed changes in the bulk behavior (mudline and L-curve) of sedimenting suspensions to self-weight fluidization, when the actual cause was the method used to mix the suspension.

In view of the experimental difficulties associated with these tests, particularly mixing, no fully reliable collection of batch sedimentation data is likely to exist at the present time, which is a significant shortcoming in view of the need to correctly validate numerical simulations. However, numerical simulation of the laboratory experiments yielded good results, and a black-box numerical modeling approach appears to be valid in spite of self-weight fluidization during consolidation of sediment, at least for the present case of a cohesive clay.

To the authors of this paper, it appears that self-weight fluidization will not have a significant impact on gravity sedimentation and consolidation in a coastal or estuarine environment, and the numerical model thus used in the present study is adequate for prediction of the time-dependent settling and consolidation of sediments in the presence of self-weight fluidization. From an environmental standpoint, it is not likely that self-weight fluidization alone will re-entrain contaminated sediments. However, it will cause fluid to move upward through (possibly contaminated) sediments, which could lead to further contamination of the water column if soluble contaminants are present. Changes in erosional strength due to changes in water content or gas content may be significant, (Jepsen et al., 2000) and the model used in the present
study may be useful in predicting such changes, if external fluidization is not a factor. Fluidization by external forcing (e.g. by subsurface gas or water expulsion), however, may be more significant, and remains an open case for further investigation.

2.7 Acknowledgments

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Bibliography


Chapter 3

Erosion Strength Parameters:
An Experimental Investigation
EROSION STRENGTH PARAMETERS: AN EXPERIMENTAL INVESTIGATION

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Authored by:
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3.1 Abstract

The present research examined the effects of different inorganic and organic properties, *viz.* sediment age, water content, clay/sand content, and microbial content on the erosional strength of sediments. For this purpose, 14 detailed erosional experiments were performed in the laboratory under well-controlled conditions in order to (1) isolate the role of each of the properties and (2) evaluate the cumulative effect on erosional strength of varying these inorganic and organic components.

The erosional tests were performed for both artificial and natural sediments in order to describe the response of these sediments to changes in the above inorganic and organic properties individually and cumulatively. These tests allowed the examination of an existing hypothesis stating that the erosional strength of a cohesive sediment can be deduced based on a comprehensive knowledge of its properties of composition (inorganic and organic) and fluid environment, provided that the effect of each property on erosional strength is known. The erosional strength tests were complemented by a mineralogic composition analysis via X-ray diffraction and an elemental analysis via X-ray fluorescence of the artificial and natural sediments, in order to provide a basis for comparison of those sediments. In order to test the effects of microbes on the erosional strength of artificial mud, bacteria extraction techniques were employed to isolate and cultivate a microbe from the natural sediment used in this study. The DNA from the microbe was sequenced in order to identify the genus (*Halomonas*) of the bacterium.

The results of this study show that the inorganic properties of sediment age, water content, and clay/sand content were generally predictable in their effects on erosional strength and were consistent with the findings of other
researchers. Although these inorganic properties were individually predictable in their effects on sediment erosional strength, it also became apparent that total erosional strength of a sediment cannot be represented accurately by the algebraic sum of the contribution of each of its properties. This is likely due to the interdependence of these properties.

With respect to the organic component of this research, it was concluded that accurate duplication of the biological components of a natural sediment in the laboratory is a very difficult task. Because of incomplete representation of the biological microcosm in the laboratory, a comparison between natural and artificial sediments may not be meaningful. Specifically, it was found that although organics were found to significantly increase the erosional strength of the natural sediment used in this study, the monoculture of Halomonas microbes extracted from that sediment and added to a pure clay decreased the strength of the pure clay by preventing soil formation. Rheological tests confirmed this observation. It is probable that other organisms present in the natural sediment contributed to its erosional strength, thereby offsetting the effect of the Halomonas microbe.

## 3.2 Introduction

### 3.2.1 Motivation

An understanding of sediment transport processes is fundamental to predicting short-term and long-term effects of changes in climate or management on the sedimentary or geomorphologic features of a region, such as a coastline, estuary, or river. Many models exist to predict sediment transport, but as Toorman (2001), points out, in many cases those models are for a par-
ticular case and location, and lead to inaccurate predictions when generalized. The next step in modeling the overall process of entrainment, transport, and deposition is to examine the fundamental principles of each process, verify those principles, and use them as building blocks to form an integrated model. In particular, transport of cohesive sediment remains a largely unsolved problem, due to the many physicochemical and biological factors influencing the “cohesiveness” of the sediment (Le Hir et al., 1993; Raudkivi, 1998; Toorman, 2001; van Ledden, 2003). Further, there are different characterizations of the strength of cohesive sediments; in general, these may be divided into mechanical and erosional strengths (Partheniades, 1965). Various methods exist for measuring the mechanical shear strength of soils; among these are the fall cone, Torvane, and direct shear tests from geotechnical engineering (Tan et al., 1994). However, such mechanical methods are often a poor predictor of the erosional strength of sediments, as they can only measure en masse failure, rather than detachment of flocs from a sediment bed; Zreik et al. (1998) and Kamphuis and Hall (1983) showed that mechanical shear strength may be one to three orders of magnitude higher than erosional strength for cohesive sediments. Determination of the threshold (critical stress) condition and the subsequent erosional behavior of cohesive sediments is the focus of the present study.

3.2.2 Previous work

The erosional strength of a soil, whether marine, estuarine, lacustrine, or riverine mud, has been measured using annular or straight flumes (Parchure and Mehta, 1985; Mitchener and Torfs, 1996; McNeil et al., 1996; Zreik et al., 1998; Hilldale, 2001; Aberle et al., 2003; Roberts et al., 2003), a rotating cylinder (Arulanandan et al., 1975), and a specialized jet device (Tolhurst et al.,
1999). Tests have been conducted in situ and ex situ, and researchers have compared effects of compaction, clay/sand proportions, clay type, organic content, pH, salinity, flora and fauna, and various other chemical and compositional parameters. As the correct value of critical shear stress $\tau_{cr}$ is strongly dependent on the physicochemical and biological characteristics of sediments, it is difficult to measure or predict for a given sediment. Typically, the sediment erosion rate is plotted as a function of flow-induced shear stress, and $\tau_{cr}$ is obtained by extrapolating the erosion rate trend back to its intercept of the shear stress axis; this intercept is the critical stress value (Parchure and Mehta, 1985; Vermeyen, 1995; Dennett et al., 1998; Houwing, 1999; Ravisangar et al., 2001).

### 3.2.3 Properties Affecting Erosional Strength

Specific trends have been observed in many previous studies. For instance, more compacted samples have higher strength (Vermeyen, 1995), as do samples containing a higher sand percentage within certain limits (Mitchener and Torfs, 1996). The history of the sediment is also significant, with sediment layers showing increased strength after aging on the order of a week; this has been variously attributed to density stratification of sediment, compaction or decreased water content, and thixotropy (Parchure and Mehta, 1985; Vermeyen, 1995; McNeil et al., 1996; Zreik et al., 1998; Wang, 2003). The pH dependence of clays demonstrates their sensitivity to environmental changes, as Ravisangar et al. (2001) have shown that erosional strength is high at low or high pH values, but strength is low at neutral pH, presumably due to the change in orientation of the clay plates (van Olphen, 1977). In general, organics (flora and fauna) have been found to increase strength as well (Parchure and
Mehta, 1985; Black et al., 2002), but work by Montague et al. (1993) showed that an excess of organics produced a lower erosional strength in a mud sample, while Dennett et al. (1998) showed that critical stress of kaolinite decreased with increasing natural organic matter (e.g. manure). Recently de Deckere et al. (2001) showed that macrofauna (soil animals longer than 1000 µm) can destabilize sediments by up to 300%, while others (Prochnow et al., 2000; Friend et al., 2003; Lelieveld et al., 2003) indicate that microbes may act to stabilize sediments through EPS (extracellular polymeric substances) secretions and growth of filaments.

3.2.4 Current Hypothesis

It has been hypothesized in the literature that the erosional strength of a cohesive sediment can be deduced based on a comprehensive knowledge of its properties of composition (inorganic and organic) and fluid environment, provided that the effect of each property on erosional strength is known. For instance, while it might seem intuitively obvious that properties which individually act to increase the strength of a sediment will act in an additive manner when combined, the result of such a combination or of a combination of strength-increasing and strength-reducing agents is actually unclear. The following experiments will test this hypothesis, using a variety of sediment samples in a controlled laboratory environment. The effects of water content, sand content, organics, sediment age, and soil microbes will be isolated by performing controlled experiments at specified initial conditions, using a pure kaolinite clay and a sand of known composition. Complementary experiments using a natural sediment sample containing these components will also be performed, and a qualitative comparison made between the results of the artificial
and natural mud cases, in order to evaluate the cumulative effects of all properties on erosional strength and to examine whether predictions of erosional strength for artificial sediments can be extrapolated to natural sediments. It is anticipated that the examination of this hypothesis will also provide some qualitative information about the relative significance of inorganic and organic components on erosional strength, and about the possible role of microscale properties on sediment erosional strength.

3.3 Experimental Setup and Procedure

3.3.1 Equipment

The experiments were conducted in the R. L. Albrook Hydraulics Laboratory of Washington State University. A tilting flume (Figure 3.1) was modified by placing artificial roughness on the bed upstream and downstream of the test section. A honeycomb ensured rectilinear flow, flow rate was controlled by a gate valve, and an instream tail gate provided uniform flow control. The tilt of the flume was controlled by an electric motor, and slope was measured directly in percent using a digital level mounted on the flume. Sampling tubes (3 mm ID) upstream and downstream of the test section were used to sample suspended sediment by siphoning the flow into polyethylene bottles. A single tube was used upstream, as the incoming flow was judged to have a laterally uniform sediment concentration due to mixing; two tubes were used for sampling downstream, in order to average out lateral variations in erosion, and all tubes were located at the same depth in the water column. The sample tray itself was a removable plastic box with dimensions given in the figure, and was easily removable from the test section of the flume for sample emplacement.
Sandpaper roughness around the top edges of the test section box minimized the effects of the flow transition from the flume floor to the sediment surface.

Figure 3.1: Schematic of tilting flume and experimental setup for erosional strength tests

A Fann 35A/SR12 viscometer was used to characterize mixtures whose erosional strength was too low to measure in the flume. This unit provides the rheological stress vs. strain rate relationship for a fluid when used in the traditional manner (Jogun and Zukoski, 1996; de Brouwer et al., 2002; Schatzmann et al., 2003). Fluid is placed in a cup, and a layer of fluid transfers stress through a gap between a variable-speed, motor-driven cylindrical shell and a concentric solid inner bob; the strain motion induced on the spring-loaded inner bob is measured directly on an indicator, and the stress or viscosity is
then computed from a table supplied by the manufacturer.

### 3.3.2 Sediments Tested

Several sediment samples of both artificial and natural muds were used for erosional strength testing. A pure kaolinite (Hydrite PX, Georgia Kaolin Co.) with a median diameter of 1.4 µm (measured using a Mastersizer, Malvern Instruments Ltd., Worcestershire UK) and specific gravity of 2.6 was used for initial testing. Kaolinite is a very common 1:1 clay mineral consisting of layered tetrahedral (typically silica) and octahedral (typically alumina) sheets, which are bonded by hydrogen bonds between hydroxyl and oxygen ions of adjacent layers, as well as van der Waals attractive forces. Due to the strength of these bonds, kaolinite is a stable clay mineral with low cation exchange capacity (CEC) and surface area (van Olphen, 1977; McBride, 1994). Further, since kaolinite layers do not separate except in “extremely polar solvents,” according to McBride (1994), it should give well-defined, repeatable results for erosion testing.

In order to examine a more realistic mud, composed of materials other than clay, the kaolinite was mixed with a calcareous sand (median diameter of 0.674 mm, specific gravity 2.6) from Puuiki Beach on Oahu, Hawaii (Smith and Cheung, 2002). A marine mud from the Adriatic Sea (near the mouth of the Po River, Italy) was also obtained via the Office of Naval Research EuroSTRATAFORM project, ([http://www.onr.navy.mil/sci_tech/ocean/321_sensing/prog_cg.htm](http://www.onr.navy.mil/sci_tech/ocean/321_sensing/prog_cg.htm)) in the form of core samples and grab samples. A particle size analysis performed by Geoscience Laboratories (Sudbury, Ontario) revealed this mud to be composed of approximately 3% clay, 55% silt, 30% very fine sand, 10% fine sand, and a remainder of other sands.
3.3.3 Procedures

Sediment preparation

In order to investigate the previously discussed strength parameters, including time of age of sediment, water content, microbe content, and sand content, the sediment samples were prepared for testing as in Table 3.1. “Sample” designates the test, “Composition” is the material used to make the erosion sample, “$\rho_{\text{initial}}$” gives the initial density of the sample as solids mass per total volume, and “Preparation” refers to the methods used to prepare the sample for testing. Of the methods, “Sedimented” refers to a process of sedimentation, wherein the water and sediment were thoroughly mixed with a high-speed rotary mixer, then allowed to settle and/or consolidate for the time period noted; “Mixed” in Table 3.1 refers to mixing the sediment and water by hand with a spatula. “Disturbed” samples were field samples which all experienced some physical disturbance during transportation, and whose bulk properties of water content and composition were additionally preserved or altered as noted in Table 3.1. Samples PK1, PK2, and PK5 were mixed thoroughly with a rotary mixer and allowed to settle into the sample tray (Figure 3.1) for 8 days, 21 days, and 1 day, respectively, from an initial height of 250 mm at the initial solids concentration noted in Table 3.1, in order to test the effects of age. Typical values assumed for sediment age are 3–6 days for numerical models (Ziegler and Lick, 1986). In the present study, density scans were conducted using the gamma-ray density profiling system described in Maxwell et al. (2003), in order to ensure equilibrium was reached in sediment compaction, by measuring density variation in the vertical direction for the duration of sedimentation. Samples PK3, PK4, KS1, KS2, and KS3 were prepared in order to test the effects of water content (PK tests) and sand con-

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<table>
<thead>
<tr>
<th>Sample</th>
<th>Composition</th>
<th>(\rho_{\text{initial}})</th>
<th>Preparation</th>
</tr>
</thead>
<tbody>
<tr>
<td>PK1</td>
<td>Kaolin</td>
<td>100 g/L</td>
<td>Sedimented 8 days</td>
</tr>
<tr>
<td>PK2</td>
<td>Kaolin</td>
<td>250 g/L</td>
<td>Sedimented 21 days</td>
</tr>
<tr>
<td>PK3</td>
<td>Kaolin</td>
<td></td>
<td>Stiff mud. Mixed with water, soaked 2 hours</td>
</tr>
<tr>
<td>PK4</td>
<td>Kaolin</td>
<td></td>
<td>Stiff mud. Mixed with water, soaked 2 hours</td>
</tr>
<tr>
<td>PK5</td>
<td>Kaolin</td>
<td>250 g/L</td>
<td>Sedimented 1 day</td>
</tr>
<tr>
<td>KS1</td>
<td>60% sand 40% kaolin</td>
<td></td>
<td>Stiff mud. Mixed dry clay and sand, soaked 21 days</td>
</tr>
<tr>
<td>KS2</td>
<td>40% sand 60% kaolin</td>
<td></td>
<td>Sloppy mud. Mixed with water, soaked 2 hours</td>
</tr>
<tr>
<td>KS3</td>
<td>40% sand 60% kaolin</td>
<td></td>
<td>Stiff mud. Mixed with water, soaked 2 hours</td>
</tr>
<tr>
<td>MK1</td>
<td>Kaolin + antibiotics</td>
<td>250 g/L</td>
<td>Sedimented 1 day</td>
</tr>
<tr>
<td>MK2</td>
<td>Kaolin + microbes</td>
<td>250 g/L</td>
<td>Sedimented 1 day</td>
</tr>
<tr>
<td>AD1</td>
<td>Adriatic core</td>
<td></td>
<td>Disturbed, initial properties preserved, soaked in growth media</td>
</tr>
<tr>
<td>AD2</td>
<td>Adriatic grab sample</td>
<td></td>
<td>Disturbed, initial properties preserved</td>
</tr>
<tr>
<td>AD3</td>
<td>Adriatic grab sample</td>
<td></td>
<td>Disturbed, dried, remixed with tap water; initial properties altered</td>
</tr>
<tr>
<td>AD4</td>
<td>Adriatic grab sample</td>
<td></td>
<td>Re-used AD4; dried, autoclaved, remixed with tap water; initial properties altered</td>
</tr>
</tbody>
</table>

Table 3.1: List of samples tested, with composition and preparation type. For samples prepared by sedimentation, the initial solids concentration is listed.
tent (KS tests); each of these samples was thoroughly mixed using a spatula, and placed in the sample tray with the specific conditions noted in Table 3.1.

In order to test the effect of microbes on the erosional strength of pure kaolinite, samples MK1 and MK2 were prepared at 250 g/L solids concentration, identically to PK5. A 1/4 strength tryptic soy broth was used as the liquid component of tests MK1 and MK2 in order to provide a growth medium for the microbes, and a low dose of antibiotics to restrict bacterial growth was added to MK1 as a control; these antibiotics included Cycloheximide (600 mg), Nystatin (10 mg), Nalidixic Acid (1 mg), and Rifampicin (1 mg). The bacteria were obtained from an Adriatic Sea sediment grab sample (see Sediments Tested) by isolation on tryptic soy agar (Becton Dickinson, Sparks, MD). A single type of bacteria that grew quickly and produced mucigel was used in this study. The bacterium was grown in tryptic soy broth to a density of 109 colony-forming units (CFU) per mL prior to inoculating MK2 with \(1.7 \times 10^8\) CFU of the microbe in suspension. Both samples MK1 and MK2 were aerated for several days with a peristaltic pump in order to provide oxygen for the bacteria in MK2 and preserve identical physical conditions in MK1. In order to characterize the microbe, cell DNA from 5 mL cultures of the Adriatic bacterium was obtained using the Qiagen QIAmp Blood DNA Mini Kit and outlined protocol. Primers specific to bacterial 16S rDNA were used (primer 8F [5'-AGAGTTTGATCCTGGCTCAG-3'] and primer 1525R [5'-AGGAGGTGATCCAGCC-3']) to amplify DNA by PCR (polymerase chain reaction). DNA was separated by gel electrophoresis and purified using an Amersham GFX purification kit. Sequencing was performed by the DNA Facility at the University of Iowa, and the bacterium appears to be of the genus *Halomonas*, which tend to be salt-loving marine bacteria (Sass et al., 2001; Béjar et al., 1998).
The Adriatic specimens AD1 and AD2 did not require mixing, as they were removed from a core tube and grab sample bag, respectively, and tested at their original water contents. Sample AD1 was soaked in tryptic soy broth for several days in order to promote bacterial growth, as in the case of MK1 and MK2. In order to directly evaluate the influence of organics on the Adriatic sediment, grab sample AD3 was dried at 105°C and remixed to approximately the same water content as AD1 and AD2; after testing the erosional strength of AD3, it was renamed AD4, and again dried at 105°C and subsequently autoclaved at 120°C and 221 psi to kill organics. AD4 was then remixed to approximately the same water content as AD1 and AD2, and tested for erosion. All specimens were prepared with tap water (pH 7-7.5) except MK1, MK2, and AD1.

Erosion testing

In order to determine the sediment erosion vs. applied fluid stress relation for a given sediment sample, tests were conducted in the tilting flume (Figure 3.1). The reservoir of the flume was flushed and filled with tap water prior to starting each experiment, the flume was set to the proper slope, and the sample tray was placed in position. As a precursor to testing, a low-flow discharge was supplied into the flume for 10 minutes in order to eliminate loose particles from the flume and sample surfaces as a source of error. The flow was gradually increased, using the valve, and careful attention was given to reaching and maintaining uniform flow. Using a similar setup, Dennett et al. (1998) found that the uniform flow, average bed shear stress equation given by

\[ \tau = \rho g H S \]  

(3.1)
was equivalent to the value obtained by measuring the velocity profile; adopting their result, the only required measurements were flow depth $H$ and slope $S$ of the bed for uniform flow (Hilldale, 2001). When the required depth was reached, the sampling tubes were opened and allowed to run until the water from the flume reached the tube outlets. At this time, the tubes were placed in the sampling bottles simultaneously for 30 seconds, collecting approximately 250 mL of water and suspended sediment in each bottle. This procedure was carefully repeated for predetermined stress increments, in order to record the average amount of sediment eroded in a 30 s period per stress increment. After all stress increments had been completed for a given sample, the suspended sediment concentration in each bottle was measured by filtering a 100 mL aliquot through a pre-weighed and oven-dried Whatman 934-AH glass microfiber filter (1 µm), which was then oven-dried at 105°C until a constant weight was reached. All weight measurements were made with a four-digit precision balance. Sediment eroded was taken as the difference between the suspended sediment concentration measured at the upstream tube and the average suspended sediment concentration measured at the downstream sampling tubes.

The typical duration of an experiment with a particular sediment sample was three days, including sample preparation, erosion testing, sampling, and suspended sediment analysis. As each data point from the flume tests required filtering, weighing, and drying several suspended sediment samples, using forceps to handle the filters, this was a very time-consuming process in itself. Tests using the gamma density profiling system had an additional layer of complexity added to the process, as did the tests using microbes.
3.4 Results and Discussion

3.4.1 Sediment Analysis

In order to provide a thorough reference for sediment composition, an elemental analysis was conducted at the GeoAnalytical Laboratory of Washington State University (Pullman, Washington) using a Rigaku automated X-ray fluorescence (XRF) spectrometer for three replicates of each specimen. Results are shown in Table 3.2. PK2(pre) was a sample of PK2 prior to flume testing, and PK2(post) was a sample of PK2 taken after flume testing; Puuiki was a sample of the calcareous sand, while AD1 and AD2 were samples of their respective tests. The Puuiki sand was predominantly calcite (CaO), with a high level of strontium (Sr). Similarly, the Adriatic specimens also showed high calcite and correspondingly high strontium levels (0.5%); Sr is often associated with calcite, dolomite, and gypsum, due to its natural sulphate form of celestine. Similar XRF results were obtained by Calanchi et al. (1996), who examined a variety of core samples from the Adriatic; they grouped Si, Al, Fe, K, Rb, Ba, Zn, V, and Ga as generally associated with silicates (e.g. quartz, illite, mica). The high values of Ni and Cr in the Adriatic specimens were most likely due to the abundance of basic rocks (e.g. basalt) in the Po River basin, according to Davide et al. (2003) who noticed levels of Ni and Cr in Po sediments which exceeded threshold toxicity concentrations. Due to the low bioavailability of these materials, they concluded that Cr and Ni in the Po River are natural in origin, rather than anthropogenic. It is likely that the Cr and Ni levels in samples AD1 and AD2 represent the composition of the sand fraction of these samples, which came from the mouth of the Po River. The pure kaolinite was rather simple, predominantly SiO$_2$ and Al$_2$O$_3$ as expected.
<table>
<thead>
<tr>
<th></th>
<th>wt % PK2 (pre)</th>
<th>PK2 (post)</th>
<th>Puiiki</th>
<th>AD1 (core)</th>
<th>AD2 (grab)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
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<td>52.47</td>
<td>1.21</td>
<td>57.16</td>
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<td>TiO₂</td>
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<td>0.677</td>
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<td>0.62</td>
<td>0.45</td>
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<td>0.000</td>
<td>0.020</td>
<td>0.12</td>
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<tr>
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<td>0.07</td>
<td>90.04</td>
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<tr>
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<td>0.24</td>
<td>7.05</td>
<td>4.75</td>
<td>5.23</td>
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<tr>
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<td>0.12</td>
<td>0.01</td>
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<tr>
<td>Na₂O</td>
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<td>2.38</td>
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<td>0.198</td>
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<td>99.50</td>
<td>99.83</td>
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<td>35</td>
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<td>113.29</td>
<td>111</td>
<td>4</td>
<td>191</td>
<td>233</td>
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<td>5.96</td>
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<td>1.5</td>
<td>14</td>
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</tr>
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<td>1</td>
<td>24</td>
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<td>39</td>
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<td>153</td>
<td>177</td>
</tr>
<tr>
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<td>33</td>
<td>0</td>
<td>2</td>
<td>7</td>
</tr>
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<td>La</td>
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<td>51</td>
<td>14</td>
<td>33</td>
<td>40</td>
</tr>
<tr>
<td>Ce</td>
<td>96.74</td>
<td>100</td>
<td>31</td>
<td>72</td>
<td>61</td>
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<td>27.49</td>
<td>26</td>
<td>1</td>
<td>11</td>
<td>13</td>
</tr>
<tr>
<td>sum tr.</td>
<td>882.62</td>
<td>877</td>
<td>5035</td>
<td>1696</td>
<td>1843</td>
</tr>
<tr>
<td>in %</td>
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<td>0.09</td>
<td>0.50</td>
<td>0.17</td>
<td>0.18</td>
</tr>
<tr>
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<td>100.00</td>
<td>100.00</td>
<td>100.00</td>
<td>100.00</td>
<td>100.00</td>
</tr>
</tbody>
</table>

Table 3.2: Results of XRF analysis of sediment samples, averaged. The upper group is percentage by weight of oxides, lower group is parts per million of trace elements.
for an aluminosilicate, with some trace metals (0.09%).

A portion of the Adriatic grab sample AD3 was analyzed for clay mineral content using X-ray diffraction (XRD) by Geoscience Laboratories (Sudbury, Ontario). The clay-sized fraction (<2 μm) was separated from a split of the sample to isolate the phyllosilicate (clay) component. Mg$^{2+}$ and K$^+$ cation saturations were then performed on splits of the <2 μm fraction, and smear mounts were prepared and analyzed by XRD. The Mg$^{2+}$ mount was exposed to glycol, then with the shift in low angle peak observed, glycerol solvation. The K$^+$ was heated to 550°C for one hour, and after each treatment the mounts were analyzed by XRD for a total of five analyses. Standards of regularly occurring phyllosilicate minerals were also prepared along with the Adriatic samples, in order to test for quality of separations and treatments, as well as instrument conditions. All separations and treatments were performed following the Geoscience Laboratories standard procedure for phyllosilicate mineral identification. The results of the XRD analyses determined that kaolinite was present in the sample. Further, an expandable phyllosilicate (clay) component, most likely smectite, was present, and chlorite was also detected. Mica was observed in the sample, as well as non-phyllosilicate minerals of feldspar and quartz; this is to be expected, with the high percentage of sand and silt present in the samples, and confirms the preceding interpretation of the XRF results.

3.4.2 Erosion Tests

A summary of the erosion test results is presented in Table 3.3, where “Age” is the sample age (as tested), “Water content” is the measured or estimated water content (defined as water mass divided by solids mass), and “$\tau_{cr}$” is the critical shear stress. Critical shear stress was estimated by projecting the
<table>
<thead>
<tr>
<th>Sample</th>
<th>Age (days)</th>
<th>Water content (final)</th>
<th>$\tau_{cr}$ (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PK1</td>
<td>8</td>
<td>173%</td>
<td>0.5</td>
</tr>
<tr>
<td>PK2</td>
<td>21</td>
<td>162%</td>
<td>3.5</td>
</tr>
<tr>
<td>PK3</td>
<td>0.1</td>
<td>85%</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PK4</td>
<td>0.1</td>
<td>81%</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PK5</td>
<td>1</td>
<td>182%</td>
<td>0.1</td>
</tr>
<tr>
<td>KS1</td>
<td>21</td>
<td>–</td>
<td>7.5</td>
</tr>
<tr>
<td>KS2</td>
<td>0.1</td>
<td>–</td>
<td>0.2</td>
</tr>
<tr>
<td>KS3</td>
<td>0.1</td>
<td>49%</td>
<td>8</td>
</tr>
<tr>
<td>MK1</td>
<td>N/A</td>
<td>360%</td>
<td>N/A</td>
</tr>
<tr>
<td>MK2</td>
<td>N/A</td>
<td>360%</td>
<td>N/A</td>
</tr>
<tr>
<td>AD1</td>
<td>N/A</td>
<td>70%</td>
<td>1</td>
</tr>
<tr>
<td>AD2</td>
<td>N/A</td>
<td>78%</td>
<td>1</td>
</tr>
<tr>
<td>AD3</td>
<td>1</td>
<td>64%</td>
<td>&lt;0.5</td>
</tr>
<tr>
<td>AD4</td>
<td>1</td>
<td>65%</td>
<td>&lt;0.5</td>
</tr>
</tbody>
</table>

Table 3.3: Summary of erosion testing results. Values in italics were estimated using measured values, dashed values were not measured, and N/A is used for measurements that were not applicable to a particular case.
erosion versus shear stress trend back to its horizontal-axis intercept; where a unique value was not clearly evident, the upper threshold limit is given as an inequality.

Effects of sediment age

In order to evaluate the effects of sediment age on erosional strength, samples PK1, PK2, and PK5 were sedimented directly into the sample tray for 8 days, 21 days, and 1 day, respectively. The previously mentioned gamma profiling system was used to measure the sediment density of PK1 and PK2 non-intrusively at 20-minute intervals over the entire sedimentation period, and Figures 3.2(a) and 3.2(b) show the sediment density measured by the gamma system as contour lines of constant density over the height of the sediment column as a function of time. PK1 and PK2 were taken from the lower 25 mm of the sediment bed in order to minimize the effects of density stratification on erosional strength; PK5 had a uniform density after 1 day, according to measurements made with the gamma system.

In Figure 3.3, the sediment eroded from samples PK1, PK2, and PK5 during flume testing is plotted as a function of the flow shear stress increment, calculated from Equation 3.1. As these samples had similar water content and identical composition, their primary difference was age (Table 3.3). The “youngest” sample, PK5, exhibited a linear erosion rate increase as shear stress increased, and had the lowest critical stress (0.1 Pa) of the samples in this comparison, as expected. The next older sample, PK1, had a critical stress of 0.5 Pa after 8 days of aging, and its erosion rate likewise appeared to increase linearly with flow stress. Upon careful examination of the erosion rate trends in Figure 3.3, PK1 appears to have had a slightly higher erosion resistance
Figure 3.2: Gravity sedimentation of pure kaolinite. Contour lines represent sediment density in g/L.

(a) Initial concentration of 100 g/L in suspension. (b) Initial concentration of 250 g/L in suspension.

Figure 3.3: Comparison of the erosion resistance of normally consolidated kaolin clay samples PK1 and PK2 with unconsolidated sample PK5.
than PK5 as shear stress increased above the critical values. PK2 exhibited higher strength than PK1 or PK5, with a critical stress of 3.5 Pa, and a lower rate of erosion overall. Evidently the aging of PK2 provided greater resistance to erosion, which is similar to observations made by Zreik et al. (1998), as well; they observed a similar strength increase with age in otherwise identical samples, and attributed that strength increase to thixotropic hardening of the clay. According to Barnes (1997), thixotropy is a restructuring of a material over time on a microscopic scale; for a clay material, this would be a rearrangement of the microfabric of plates into a more ordered (and stronger) fashion. Although there were slight differences in the water content of the samples, the results of the present study clearly indicate that age is a factor apart from water content. Further, sediment microstructure (thixotropy) appears to play a dominant role in determining the critical stress and overall erodibility of a sediment sample, and its effect is not necessarily proportional to the typical parameters of sediment water content or bulk density.

The role of water content

The effects of water content on erosional strength are discussed in this section. Samples PK3 and PK4 were prepared at 85% and 81% water content respectively, using identical methods in order to compare repeatability of erosional strength results in case of inhomogeneities from the mixing process (see Sediment preparation). Each sample was mixed thoroughly at the predetermined water content and placed in the sample tray, which was then soaked in water for two hours prior to testing. Sample PK5, with a water content of 182%, was prepared by sedimentation of a well-mixed suspension over a 24-hour period, and was not subject to inhomogeneities.
Figure 3.4: Comparison of the erosion resistance of pure kaolin samples PK3 and PK4 (lower water content) with PK5

In Figure 3.4, the results of the erosion tests of PK3, PK4, and PK5 are shown for comparison; as discussed previously, PK5 has a linear erosion rate and is relatively weak. Samples PK3 and PK4 both exhibited a much lower erosion rate than previously tested pure kaolin samples. Although it had a slightly lower water content than PK3, PK4 has the same erosion rate, implying that a small change in water content (4%) is insignificant; in contrast, the much larger difference in water content between PK5 and PK3 or PK4 indicates clearly that high water content leads to lower erosional strength. The experiments of McNeil et al. (1996) showed that erosion rate was not sensitive to water contents ranging from 50-80% for silty materials; Houwing (1999) examined the critical stress and erosion rate of intertidal mud, and found no significant relation between erosional strength and water content over a water content range of 40-120%, but the strength in the study of Houwing (1999) may also have been affected by sand content and benthic diatoms. PK3 and PK4 show higher resistance to erosion than the muds tested by Houwing (1999),...
which had a critical stress of 0.11-0.18 Pa; PK3 and PK4 exhibit strength comparable to the riverine silt mud from the Detroit River, (MI) and Lower Fox River, (WI) tested by McNeil et al. (1996) ($0.1 \leq \tau_{cr} \leq 5$ Pa) or the cohesive river sediment tested by Vermeyen (1995) from the Rio Grande River, (TX) at $4.8 \leq \tau_{cr} \leq 6$ Pa).

It was not possible to subject samples PK3 and PK4 to the full erosion stress capability of the flume ($\sim 14$ Pa), as PK3 failed abruptly when a piece of the sample broke away en masse and lodged against the downstream sampling tubes (Figure 3.5). As PK4 was prepared identically to PK3, it was expected that PK4 would fail in the same manner; as a consequence, PK4 was not tested to as high a shear stress as PK3, in order to avoid damage to the sampling tubes. Although a clear value for the critical stress of PK3 and PK4 is not apparent in Figure 3.4, it is judged to be $\tau_{cr} \leq 1$ Pa. Samples PK3 and PK4 exhibited critical strength similar to that of sample PK2, the 21-day sedimented sample of the previous section; in the photograph of PK3 (Figure 3.5), the surface of the sediment is essentially unchanged from its initial state, apart from the hole left by the failure on the left-hand side of the photo. It is suggested here that insufficient time (0.1 days) was allowed for thixotropy to have an effect throughout the samples, and that the underlying layer bonds were therefore not as well-developed as those of PK2.

**Comparisons of clay-sand mixtures**

In order to assess the role of sediment composition on erosional strength, samples KS1, KS2, and KS3 were prepared. Sample KS1 was a mixture of 60% Puuiki calcareous sand and 40% kaolin by weight; KS2 and KS3 were composed of 40% Puuiki sand and 60% kaolin by weight.
A comparison of the eroded sediment as a function of shear stress for samples KS1, KS2, KS3, and HIL is provided in Figure 3.6. The HIL sample was composed of 60% sand, 37% clay+silt, and organics (roots), and was obtained from the loess banks of Union Flat Creek in the Palouse region of Washington, USA. According to Hilldale and Papanicolaou (2001), the HIL sample had a critical stress of 5.5 Pa. KS1 and KS3 had similar critical values to the HIL sample (Table 3.3), but have higher erosion rates overall, likely due to organics in the HIL sample and its greater age (McNeil et al., 1996). KS3 had a water content of 49%, close to that of the HIL sample at 55%, and KS1 appeared to have a similar water content, based on visual observation. Although sample KS2 has the same composition as KS3, it displayed a higher erosion rate than the other samples in this series, and had a critical stress value of 0.2 Pa. This weakness is attributed to the higher water content of the sample, again based on visual observation.

While the critical stress values of KS1 and KS3 can not be directly
Figure 3.6: Comparison of the erosion resistance of kaolin-sand mixtures KS1, KS2, and KS3 with the silt mud (HIL) tested by Hilldale and Papanicolaou (2001)

compared to those of PK3 and PK4 due to the higher water content of PK3 and PK4, if one considers the previously discussed insensitivity of erosional strength to water content variations in the 50–80% range, it appears that in general a mud composed of clay and sand is indeed stronger than a purely cohesive material. An analogy might be made to the relative strengths of pure cement powder and cement+aggregate (concrete). The cohesive sediment apparently provides a bridge between the more rigid sand grains, and although some fines may erode at low stresses, it is clear from Table 3.3 that clay-sand mixtures exhibit strong behavior overall. The observations of this section are consistent with reports by other researchers, (e.g. Mitchener and Torfs, 1996), and it is evident from the results presented so far in this study that critical stress alone is not a sufficient metric to judge the erosional strength of a sediment, just as density or water content is not necessarily directly proportional
to the age of a sediment.

Comparison of samples containing microbes

Samples MK1 and MK2 were prepared in order to examine the possible effects of the microbe culture from the Adriatic Sea on pure kaolinite, autoclaved at 120°C and 221 psi to kill organics. MK1 was used as the control case, at a solids concentration of 250 g/L kaolin in the soy broth growth media, and with antibiotics added to inhibit bacterial growth; MK2 was also prepared in soy broth at 250 g/L, but inoculated with the microbes. After allowing several days time with aeration for the microbe culture to grow, both MK1 and MK2 were prepared for erosion testing in the same manner as PK5 (which had the same concentration of solids) by thoroughly mixing the samples with a rotary mixer and allowing each to sediment into a sample tray for 24 hours.

After completion of the 24-hour sedimentation period, however, the sediment and water merely drained out of the sample tray joints as the tray was removed from the sedimentation tank. Visually, the kaolinite in samples MK1 and MK2 appeared to be suspended in a slurry, rather than aggregating and settling to form a soil as PK5 did. The pH of MK1 and MK2 was measured with a hand-held tester, and found to be neutral. As erosion testing was not possible in this case, due to the slurry nature of MK1 and MK2, the viscometer was used to make a quantitative comparison of the effect of microbes on the mixture rheology; various authors (e.g. de Brouwer et al., 2002; Penner and Lagaly, 2001) have utilized similar equipment for analysis of slurries. According to Macosko (1994), the gap width to particle size ratio for the concentric cylinder viscometer must be at least 10; the gap width in the present study was 1.12 mm, which is well within this criteria. Figure 3.7 compares the stress
Figure 3.7: Comparison of stress-strain curves for MK1 and MK2. Also shown are two reference mixtures of pure kaolin and deionized water at solids concentrations of 250 g/L and 125 g/L. The line marked “water” is the theoretical stress-strain relation for water.
versus rotational strain rate curves of MK1 and MK2. Also shown in Figure 3.7 are curves for 125 g/L and 250 g/L mixtures of pure kaolinite and deionized water, which were mixed thoroughly and sedimented for 24 hours, then mixed again before testing. The 250 g/L pure kaolin exhibits plastic behavior, and has the highest viscosity overall; the other mixtures appear to exhibit Bingham plastic behavior (White, 1991), and have much lower stress values for a given strain (note that the upper curves are more viscous). Although the viscometer tests do not compare directly with the sediment erosion tests conducted in the flume, the viscometer does provide a measure of the gel structure of a slurry, and it is certain that slurries with a higher concentration of solids (near soil-forming conditions) will have a higher overall viscosity and plastic yield stress than those which have a lower concentration of solids (Jogun and Zukoski, 1996; Penner and Lagaly, 2001). In all cases, the viscosity was higher than that of pure water, which was calculated based on a room-temperature value of $10^{-3}$ Pa-s and also presented in Figure 3.7.

The surprising results illustrated in Figure 3.7 are that the antibiotic-kaolin mixture in growth media (MK1) has a lower viscosity than kaolin in deionized water at the same concentration of solids; further, that the microbe-kaolin mixture in growth media (MK2) has a lower viscosity than any other tests. This indicates that the microbe culture used did not have a strengthening effect, but actually weakened the floc network in the suspension. In the case of MK1, either the antibiotic killed microbes that were present in the reference tests and necessary for flocculation, or the growth media itself acted to prevent soil formation; as the growth media contained 43 mmol/L NaCl and 7.1 mmol/L potassium diphosphate ($K_2HPO_4$), both of which have the possibility of interacting chemically with the kaolinite, the latter possibility is deemed more likely. Penner and Lagaly (2001) and Jogun and Zukoski (1996)
found that sodium diphosphate (Na$_2$HPO$_4$) was a very effective dispersing agent, due to adsorption of its multivalent anion by kaolinite, and it is likely that the potassium diphosphate used in the present study had a similar effect. Penner and Lagaly (2001) also noted that “...increased particle mobility due to compression of the diffuse ionic layer is also important...”, as the anions recharge the kaolin edges and cancel the edge/face interaction; this recharge is also accompanied by an increase in pH, which is initially acidic in the case of pure kaolin mixed with distilled water (Michaels and Bolger, 1962). The air injected into the system for aeration (see Sediment preparation) could also inhibit flocculation (Montague et al., 1993; Prochnow et al., 2000), but this is considered unlikely in the present case, as the air pipette only produced bubbles over a small area.

The effect of the microbes in the present case is poorly understood. According to Béjar et al. (1998), several strains of the species *Halomonas eurihalina* examined by them produced only one type of EPS, an anionic polysaccharide. Béjar et al. (1998) examined the rheology of this EPS in comparison with distilled water, and found an increase in viscosity under acidic conditions, but very little change under neutral conditions. Arias et al. (2003) considered aqueous solutions of mauran, an anionic EPS produced by a certain strain of the halophilic bacteria *Halomonas maura*. They found that these solutions were highly viscous, and exhibited pseudoplastic, viscoelastic, and thixotropic behavior over a wide pH range, and that mauran also had a high capacity for binding cations such as lead. de Brouwer et al. (2002) added EPS extracted from intertidal sediments to a fine sediment mixture, and found no change in slurry rheology, although the EPS was clearly adsorbed to the sediment. They speculated that some action of diatoms was necessary to structure the EPS in such a way that it would add strength to the sediment, and it is also possible
that additional chemical or organic components are necessary to structure the EPS, such as cations for bridging (Stal, 2003). Marshall (1986) notes that most bacteria have a size range of 0.15–2.0 μm in length or diameter, comparable to the kaolin particles in the present study. Further, bacteria possess a net negative charge (Marshall, 1986), and it is conjectured here that the microbes or their EPS could act in a similar manner to the phosphate ions mentioned previously, i.e. recharging the kaolin edges and canceling the edge-face interactions that are typically a precursor to sedimentation and soil formation (Partheniades, 1993). Although it is possible that this dispersion of the clay particles would yield a stronger sediment over time, as the particles could form a structure with smaller voids (Partheniades, 1993), other material, such as sand, may be necessary to aid in consolidation (Torfs et al., 2001, 1996).

**Adriatic sample erosion tests**

As various researchers (e.g. Mitchener and Torfs, 1996; van Kessel and Blom, 1998; Prochnow et al., 2000) have shown, it is difficult to directly compare the erosional strength of artificial and natural muds. In an effort to assess the erosional strength of combined inorganic and organic components in a natural mud, and to investigate the accuracy of approximating the erosional strength of a natural mud based on tests conducted with an artificial mud of similar properties, erosional tests were conducted on the natural sediment samples from the Adriatic Sea.

Knowing that the Adriatic samples AD1 and AD2 are well-aged, and that they contain clay, silt, sand, and organic material (see Sediment Analysis), the reader can estimate that the behavior of the Adriatic samples based on age, composition, and water content, will be comparable to that of the strongest
samples tested. Considering the critical erosion stress of pure kaolin sample PK1 (aged 21 days) to be 3.5 Pa, and that of the kaolin-sand mixture KS3 to be 8 Pa (based on Table 3.3), one would estimate similar trends, i.e. a high critical stress and low overall erosion rate for AD1, AD2, and AD3, with AD4 unknown. Based on the strength-reducing behavior of the microbes as discussed in the preceding section, it is expected that they will reduce the erosional strength of the sediment, although it is not possible at present to quantify the reduction.

![Figure 3.8: Comparison of the erosion resistance of Adriatic samples AD1, AD2, AD3, and AD4](image)

Sample AD1 was prepared by removing it from the coring tube, placing it in the sample tray, and soaking it in the soy broth. Sample AD2, a grab sample, was prepared by extruding it from its plastic bag into the sample tray; minimal shaping was required. Sample AD3 was prepared from a grab sample as well, by first drying it at 105°C, and then remixing to a controlled
water content approximately equal to that of AD1 and AD2 (Table 3.1); AD3 was then tested for erosion. Following the erosion test of AD3, the sample was again dried at 105°C and autoclaved at 120°C and 221 psi in order to kill organics. This autoclaved sample was renamed AD4, and tested again for erosional strength.

Examination of Figure 3.8 shows that tests AD1 and AD2 are indeed characterized by very low erosion rates, although the critical stress for both samples is estimated at 1 Pa (Table 3.3). No visible erosion occurred during either of these tests, other than small pockets which may have been inhomogeneities introduced during sample placement. Tests AD3 and AD4 show much higher erosion rates (and therefore reduced strength); extrapolation of the erosion rate would yield a negative critical stress, indicating a weak mixture. The striking decrease in strength of AD3 vs. AD1 and AD2 (in spite of the higher water contents of AD1 and AD2) is attributed to the drying process (at 105°C), which may have killed a few microbes (Ann Kennedy, personal communication), and the remixing of the sediment (which reversed the effects of sediment age). However, the autoclaving of AD4 (same physical sample as AD3) produced a further decrease in strength, which seems to indicate that the organic content of the sample was very significant, and actually acted to increase overall erosional strength. Removing the organics from AD3 produced erosional behavior comparable to the weaker samples of pure kaolinite (e.g. PK1 and PK5).

It should be noted that the result of removing the microbes from the Adriatic sediments is counter to the results of tests MK1 and MK2, indicating that other microbe cultures existing in the natural sediment may enhance sediment strength, or that a sand fraction may be necessary to enable the microbes to increase the erosional strength. Without direct visual examination of
the sediment microstructure by electron microscopy, the mechanism by which the microbes decreased viscosity is open to speculation, and this phase of the experiment underscores the present difficulty of making generalizations about the effects of organics on erosional strength.

3.5 Conclusions

This study examined the effects of different inorganic and organic properties, viz. sediment age, water content, clay/sand content, and microbial content on the erosional strength of sediments. It has been hypothesized in the literature that the erosional strength of a cohesive sediment can be deduced based on a comprehensive knowledge of its properties of composition (inorganic and organic) and fluid environment, provided that the effect of each property on erosional strength is known. In order to test this hypothesis, experiments were performed in the laboratory under well-controlled conditions in order to (1) isolate the role of each of the properties and (2) evaluate the cumulative effect on erosional strength of combining these inorganic and organic components. It was deemed important that erosional tests be performed for both artificial and natural sediments, in order to describe the response of these sediments to changes in the above properties individually and cumulatively. The results of these two tests were compared to examine whether predictions of the erosional strength of an artificial mud can be extrapolated to a natural sediment of similar composition, and whether the total erosional strength of a sediment can be estimated by algebraically summing the strength contribution of each of its components. The findings of this study can be summarized as follows:
• The bulk properties of water content, age, and sediment composition were generally predictable in their effects on erosional strength, and were consistent with the findings of other researchers. Due to the interdependence of these properties, however, it is clear that total erosional strength of a sediment cannot be represented accurately by the algebraic sum of the contribution of each of its properties.

• Although organic components were found to significantly increase the erosional strength of the Adriatic sediment, a monoculture of microbes extracted from the Adriatic sediment in the laboratory and added to a pure clay decreased the erosional strength of the clay by preventing soil formation. This contradiction is attributed to the difficulty of accurately representing a natural biological microcosm in the laboratory. In the present study, it was not possible to extract and grow all of the bacteria from the Adriatic sediments in the laboratory; hence, it is probable that other organisms present in the natural sediment contributed to its erosional strength, perhaps offsetting the strength-reducing effect of the Halomonas observed in the present study.

• Because of the apparently incomplete representation of the biological component of the natural sediments, no direct comparison could be made between the natural and artificial sediments, despite their strong similarity in bulk properties. Accurate representation of the biological components remains open. Furthermore, future studies should include visualization of the sediment microstructure at microbe-scale resolutions, using instruments such as the scanning electron microscope or X-ray computed tomographer to evaluate the different mechanisms of biological stabilization or destabilization.
3.6 Acknowledgements

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Chapter 4

Experimental Investigation of Minimum Fluidization Conditions for Kaolinite
EXPERIMENTAL INVESTIGATION OF MINIMUM FLUIDIZATION CONDITIONS FOR KAOLINITE

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TBD

Authored by:

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4.1 Abstract

The present study considers the fluidization behavior of a pure clay sediment via laboratory experiments. In order to develop an experimental methodology for determining the minimum fluidization conditions (maximum bed strength) for a kaolinite clay, a flow of water was directed vertically upward through an otherwise static bed of clay at a fixed initial volume fraction of solids. Tests were conducted at various flowrates, ranging from 2–10 mL/min. The pressure in the bed was measured at four different locations over the time of the test, and the pressure in the fluidization column base was measured simultaneously. Volume fraction of solids was measured using a gamma radiation system, and outward changes in the bed were recorded with a high-resolution digital camera for later image analysis. Failure of the bed was characterized by either horizontal cracking in the side of the sediment bed, or by a slit crack in the top of the bed, through which water flowed freely. The pore pressure at which this occurred appeared to vary with the flowrate into the sediment bed, suggesting that a unique value of pressure may not exist; however, a maximum pressure in the bed appears to exist, although further testing is suggested.

4.2 Introduction

4.2.1 Definition and Application

For the purposes of this paper, fluidization will be considered as a process which suspends particles in a bed (e.g. Crowe et al., 1998). Although fluidization may be caused by self-weight consolidation of sediment (channelling) according to various authors (e.g. Vesilind and Jones, 1993; Holdich and Butt,
1995; Papanicolaou and Maxwell, prep), the present discussion will be confined to externally forced fluidization of a sediment layer.

Fluidization has many applications in the chemical processing industry, via the fluidized bed. In general, particles are fluidized with gas or liquid, and the state of the fluidized bed varies with the flow rate of the fluid (e.g. Srivastava and Sundaresan, 2002). Environmental fluidization of a cohesive sediment will be considered here, with water as the fluidizing agent. In nature, for example, an estuarine mud may be fluidized by several means, including wave action, seismic activity, and hydrologic (subsurface) flow. According to Mehta (1989), fluidization of a cohesive bed and shearing action are both erosion processes, and Govindaraju et al. (1999) point out that it is necessary to understand the processes of sediment deposition, dewatering of fluid mud, and consolidation of sediment, concomitant to erosion.

The overall goal of this study is to develop a better understanding of the erosional resistance of cohesive sediments in natural systems, as mentioned above. To this end, (Papanicolaou and Maxwell, prep) examined the effects of self-weight fluidization on batch sedimentation of kaolinite, and found that it had little or no effect on the overall sedimentation behavior (settling curve). They also found that a maximum value of the volume fraction of solids of kaolinite under gravity sedimentation 0.19; this volume fraction was later tested by (Maxwell et al., rev), who examined the erosional strength of kaolinite clay under various depositional and flow environments. As fluidization and erosion are interrelated, the present study aims to complement those tests by examining kaolinite strength in a forced fluidization case through experimentation.
4.2.2 Critical Literature Review

Once a sediment material has settled, it becomes a static or fluid mud, depending on depth, age, and water flow characteristics (Ross and Mehta, 1985). However, Lee and Mehta (1997) note a number of problems in characterizing mud behavior, such as the fluidization potential of the mud, which is dependent on flow properties (waves) and the dissipative character of the mud itself. A number of approaches have been used for modeling the time-dependent behavior of fluid mud, including the empirical mixing models of Kranenburg and Winterwerp (1997a,b) and Jiang and Mehta (2000). The theoretical fluidization model utilized by Li and Mehta (1997) and Verbeek and Cornelisse (1997) is the Voigt viscoelastic model, while Yamamoto et al. (1978) used the Biot model (Biot, 1941) of poroelasticity. It remains an open question whether it is more correct to model the fluid mud as a viscoelastic fluid or a poroelastic solid.

The aforementioned studies considered cohesive sediments under wave fluidization. Other researchers have focused on more typical fluidized-bed studies to describe the behavior of environmental and other systems. Roche et al. (2001) used water to fluidize a bed of volcanic particles, and characterized the system via pressure measurements and sectioning the sediment bed. Nichols et al. (1994) studied the fluidization patterns of non-cohesive sediments, and also a mixture of cohesive and non-cohesive particles. M’chirgui et al. (1997) considered the pressure fluctuations in a gas-fluidized bed of particles (Geldard Group B particles, (Geldart, 1973)), with simultaneous image analysis of the bed behavior. They found that above a critical gas flow rate, the bulk behavior of the bed exhibited a regular pattern of fluctuations in pressure and height. Research conducted by Kage et al. (2000) and Lu and Li (1999) ana-
lyzed the pressure changes in gas-fluidized beds using Fast Fourier Transform and wavelet techniques, respectively, and found that bubbling behavior could be characterized by frequency analyses. Valverde et al. (2000) examined the tensile strength of a powder bed (xerographic toners) under gas fluidization, and found that the “breaking point” of the toner increased with increasing consolidation stress prior to fluidization. Duru and Guazzelli (2002) suggest that instability of voidage waves in fluidized beds of glass particles can lead to “transient buoyant blobs,” indicating that the volume fraction of solids is a key time-dependent parameter, as well as pressure.

As yet, no “failure criteria” exists for the fluidization strength of cohesive sediments, and the focus of the present study is the development of a methodology which will facilitate determination of such a strength. It is hypothesized that this occurs at a maximum pore pressure inside the sediment, as in the case of hydraulic fracture (Engelder and Lacazette, 1990; Murdoch and Slack, 2002).

### 4.3 Objectives and Methodology

The present study focuses on the external fluidization of a cohesive sediment via water. This scenario is different from many cases in the literature, in that environmental fluidization studies typically use non-cohesive particles, or fluidize the system by means of simulated wave action; traditional fluidized bed studies tend to focus on gas-fluidization, and typically consider non-cohesive particles. The additional level of complexity associated with cohesive particles necessitates careful experimentation. A dimensional analysis of the well-known Ergun equation for the pressure drop through a fluidized
bed was given by Niven (2002) as

\[
\frac{\Delta P}{L} = \Phi (\mu, \rho, U, d_p, g, \epsilon, \xi_p)
\]  

(4.1)

where \(\Delta P\) is the change in pressure over the height \(L\) of the bed, \(\mu\) is the dynamic fluid phase viscosity, \(\rho\) is the fluid density, \(U\) is the superficial fluid velocity (averaged over the bed cross-section), \(d_p\) is the diameter of the particle, \(g\) is gravity, \(\epsilon\) is the porosity of the bed, and \(\xi_p\) is the particle shape factor.

For cohesive particles, the electrochemical bonds between the particles must also be accounted for, as well as other variables such as the resting time of the sediment, its stress history, and entrained gas contents. In the present study the conditions necessary to induce fluidization were examined, with an aim towards finding a minimum flowrate for fluidization. For this purpose, a flow of water was introduced vertically upward into a bed of unconsolidated clay at varying fluid flowrates.

Based on a review of available literature (e.g. Asif et al., 1994; Nichols et al., 1994; M’chirgui et al., 1997; Kage et al., 2000; Roche et al., 2001; Duru and Guazzelli, 2002), it was determined that several key parameters should be measured:

- Sediment volume fraction of solids
- Pore fluid pressure
- Outward changes in bed structure (bed height, size of fluidization pipes, cracks)

In order to measure these parameters with as little effect on the system as possible, non-intrusive techniques were used when available.
4.3.1 Experimental Conditions

The sediment used in this study was prepared from dry kaolin powder (Hydrite PX, Georgia Kaolin Company) with typical median particle size of 0.68 µm and specific gravity of 2.58, according to the manufacturer. A specific gravity test placed the actual value at 2.63, and a value of 2.6 was used in calculations. Kaolinite is a 1:1 clay mineral consisting of layered tetrahedral (typically silica) and octahedral (typically alumina) sheets, bonded by hydrogen bonding between hydroxyl and oxygen ions of adjacent layers, as well as van der Waals attraction. Due to these bonds, kaolinite is a stable clay mineral (van Olphen, 1977), with low cation exchange capacity (CEC) and surface area (McBride, 1994). Deionized water was used in all tests, in order to eliminate potential variations in ionic strength of tap water. As it was not apparent from the literature investigation which flowrates would be suitable, the authors chose a series of flowrates (2–10 mL/min) which bracketed those used by Roche et al. (2001) in their fluidization of volcanic material. The volume fraction of solids chosen for the experiments was based on the results of batch sedimentation tests using the same material; the maximum volume fraction of solids obtained through gravity sedimentation was 0.19, and a similar volume fraction (0.17) was used in erosional strength tests conducted by the authors (Maxwell et al., rev). Consequently, as strength increases with volume fraction, this “maximum” value was deemed a good starting place for the present study. A relatively thin layer of sediment was used in order to avoid stratification effects, although according to tests by (Maxwell et al., 2003), this should not be an issue for at least the first few hours of sedimentation. Additionally, a thin layer was desired in order to ensure that pressure measurements in the layer are representative of the pressure in the entire layer.
4.3.2 Equipment and Measurements

The test column as shown in Figure 4.1 was constructed from cast acrylic tubing with an inside diameter of 140 mm and 6.35 mm wall thickness. Water was pumped into the base of this setup using a peristaltic pump with a digital speed controller. An inline pulse dampener was used to soften the pulses of the pump, and the flow from the pump passed through a series of filters (Figure 4.2) before actually reaching the clay bed. The filters used were 6.5 in. disks (KenAG, Ashland, Ohio) used for filtering milk. These filters were easily replaceable and could also be cleaned by backwashing; the primary purpose of the filter was to prevent the solids from entering the column base en masse, and they appeared to be effective for this purpose. The grooved ring in Figure 4.2, immediately above the rubber O-ring, was specially constructed in order to minimize flow up the wall of the column (short-circuiting). By requiring a more tortuous path from the inlet to the walls of the cylinder, it was hoped that this would provide more realistic results, without using a mesoscale model setup.

For tests Aug12-N–Aug18-N, a single outlet was used for the overflow.
Figure 4.2: Filtering arrangement used in the base-to-cylinder flange of the test column. 

(Figure 4.1). A second outlet was added diametrically opposite the first for the remaining tests, in order to avoid any preferential flow gradients. No visible changes resulted from this modification.

In order to facilitate the pressure measurements, pore pressure sensors were constructed, using EPXN-V01-5P pressure sensors (Entran Devices, Inc., Fairfield, NJ). The EPXN-V01-5P is a passive-resistive miniature pressure sensor, with 0–5 psig range and a nominal 50 mV full scale output. A physically larger sensor would provide greater sensitivity, but would not be usable in the space constraints of the laboratory setup, which seeks to provide “point” measurements. On advice from Entran (Jim Pierson, personal communication, 2002), the sensors were fitted with a threaded brass tip, to which was epoxied a porous metal disc; the sensor tips were then submerged in silicone oil (DMS-T12, Gelest, Inc., Tarrytown PA) inside a vacuum jar, and a high vacuum drawn over the apparatus. This replaced the air in the sensor/filter combination with silicone oil, which was held inside the porous metal filter by surface tension effects, yet provided an incompressible medium between the sensor and the pore fluid, while the porous metal supported the clay bed structure. The sensor output voltage was measured using an Agilent 34970A Data Acquisition/Switch unit, equipped with a 16-channel reed relay multiplexer for high-speed switching (Agilent 34902A). This provided 5-1/2 digit precision
voltage measurements, at a rate of approximately 10 Hz per channel for five channels. The data from the switch unit was read using a custom LabView (http://www.ni.com/) program, running on a personal computer, and later processed using Igor Pro software (http://www.wavemetrics.com/).

Images were captured simultaneously with pressure, albeit at a reduced frequency (2 Hz) because of digital image storage considerations. A Sony DFW-X700 FireWire camera equipped with an optical zoom/macro lens was used to capture images to a personal computer, at a maximum frame rate of 15 fps. The images were square-pixel images at 1024x768 pixels resolution, and high-speed capture required a significant amount of disk storage capacity. The photos were converted to grayscale, scaled to 800x600 pixels, and resampled at 72 dpi using TIFFany3 on Mac OS X. Image analysis was performed using NIH ImageJ, a Java-based image processing program, freely available from http://rsb.info.nih.gov/ij/. The images were cropped to a specific area of interest, encompassing a third or more of the sediment top, and a binary threshold was used to separate the sediment layer from the remainder of the image. Particle analysis of the image sequence was used to determine the area of the bed “particle,” which changed over the course of the test (Figure 4.3); dividing by the width of the cropped region gave the average height of the bed as a function of time. Maintaining an even level of light across the side of the bed was critical for the thresholding operation; 30-watt portable incandescent lamps were used for this purpose.

The experiments were conducted initially using only the pressure and camera measurements, in order to gain familiarity with the setup and procedures. All of the experiments were subsequently repeated at least once using a gamma radiation system (described in Maxwell et al. (2003)) to measure the volume fraction of solids in the bed over time. Briefly, the system consists of a
570 mCi $^{241}$Am gamma radiation source, which emits a monoenergetic photon at 60 keV; by calibrating the system with various mixtures of kaolinite and water at known volume fraction of solids, it is possible to obtain quite accurate measurements of the solids volume fraction with ±0.5 mm vertical accuracy. For the purposes of this study, the system was focused at a single point (the midpoint of the bed), and discrete density measurements were taken at a rate of 9 Hz, the maximum allowed by the photon counting system. The error in volume fraction of solids introduced by this short counting time was significant, and a Butterworth filter with a passband of 0–0.02 Hz was used to obtain an overall trend in the volume fraction of solids; the filter was designed and applied in software using the Igor Pro Filter Design Laboratory.

4.3.3 Experimental Procedures

Testing was conducted at the Lucille A. Carver Mississippi Riverside Environmental Research Station of the University of Iowa. The clay and deionized water were thoroughly mixed using a Jiffy wand rotary mixer (Jiffy Mixer Co., Inc., Riverside, CA) mounted on an electric drill. Papanicolaou and Maxwell (prep) found this to be an effective tool for mixing kaolinite and water. The mixture was then allowed to sit at least 24 hours before using, as Farrow
et al. (2000) showed that this was necessary in order to achieve a steady-state particle size. The clay was mixed again for five minutes prior to testing, in order to remove stress history effects and ensure a uniform volume fraction of solids (Channell et al., 2000). Preliminary testing showed that fluidization behavior was influenced by the presence of gas entrained in the clay bed; in order to minimize the effects of this gas, the clay was degassed under vacuum for 30 minutes prior to testing, at which point bubbling had ceased. After degassing, the clay pH and temperature were recorded; pH was typically 4.5–4.8, which is consistent with reports by other researchers (e.g. Michaels and Bolger, 1962). Concurrent with the clay preparation, a background pressure scan was run, pumping water through the column in order to obtain the spectral pressure characteristics of the pump and the average hydrostatic pressure. Having placed the camera and the gamma system in the correct locations, the column was carefully leveled using a spirit level in order to avoid variations in fluidization behavior as reported by Daw et al. (2000). The clay-water mixture was carefully poured into the test cylinder to a height of 30 mm, such that the pressure sensors were at the vertical midpoint of the bed. The surface of the bed was tooled flat by hand, using a spatula, before starting the pressure and volume fraction scans. After scanning for at least 30 seconds, the camera and pump were started simultaneously, and all instruments were allowed to run until termination of the test. As water rose through the bed, it was eliminated by outlets at the bed surface, in order to avoid significant accumulation of water on top of the bed over the course of a test.

A test was terminated after no visual change was observed for some time; typical test durations were 20–60 minutes, depending on the flow rate, but the bed generally showed some sign of fracture long before termination.
4.4 Results and Discussion

One of the difficulties in conducting the experiments was the lack of a criteria for fluidization; typical descriptions from fluidization literature indicate bubbling or slugging flow, whereas the present case did not exhibit this behavior. A fluidized state was considered to exist when the bed had cracked visibly, or a water release hole or slit formed in the top of the bed; these phenomena appeared to be related, in that a crack in the side of the bed (e.g. Figure 4.4) was a precursor to an opening in the top. Similar phenomena have been reported by Nichols et al. (1994) and others, for forced fluidization.

Some differences exist between this case and the self-weight fluidization considered by Papanicolaou and Maxwell (prep), however; in the self-weight fluidization cases, flow generally passed through vertical pipes in the sediment, both along the column wall and over the cross section of the column. Pipe size varied from 0.5–5 mm, and volcanoes in the top of the sediment (remnants of fluidization pipes) had an apparent size of ∼1 mm. In the present case, horizontal cracks of 0.5–3 mm typically developed in the bed, and flow was then piped to the top along the column wall or through a subsequent break in the bed surface. In some tests, vertical (sheetlike) cracks such as those described by Roche et al. (2001) formed near the middle of the sediment bed, allowing water to escape directly from the bottom of the bed. The self-weight cases generally exhibited fluidization behavior at lower sediment volume fractions, however, and the volume fraction of the present study was chosen such that it would not be affected by self-weight fluidization. Hence, some differences in sediment behavior may be expected, as the higher volume fraction should have a higher strength (Quintanilla et al., 2001; Maxwell et al., rev, e.g.). A summary of the experimental results is presented in Table 4.1. “Test” is a
Figure 4.4: Image from test Aug12-4, showing a horizontal crack extending through the sediment bed. The scale tick marks are in millimeters.

descriptor for the test, $\phi_{so}$ is the initial volume fraction of solids, $Q$ is the volumetric flowrate, $t_c$ is the time between the start of the test and the first observance of a crack, $\Delta p_c$ is the differential pressure between the base sensor and the average value of the bed sensors at time $t_c$, $\bar{p}_{base}$ is the time-averaged pressure in the base, $\bar{p}_{bed}$ is the time and space-averaged pressure in the bed. In preliminary tests with “high” flowrates (20–80 mL/min), the entire bed was lifted up as a plug, until a water escape structure formed (usually along the side of the plug); the flowrates chosen for the tests in Table 4.1 did not have this problem. Other preliminary tests revealed the importance of removing entrapped air from the column base beneath the filters, as well as degassing the clay/water mixture with vacuum; gas bubbles would otherwise propagate through the clay bed and eject from the surface, implying a three-phase flow condition.
Table 4.1: Summary of tests performed and results.

<table>
<thead>
<tr>
<th>Test</th>
<th>( \phi_{so} )</th>
<th>( Q ) (mL/min)</th>
<th>( t_c ) (seconds)</th>
<th>( \Delta p_c ) (psi)</th>
<th>( \bar{p}_{base} ) (psi)</th>
<th>( \bar{p}_{bed} ) (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aug12-2</td>
<td>0.19</td>
<td>2</td>
<td>240</td>
<td>N/A</td>
<td>N/A</td>
<td>0.02093</td>
</tr>
<tr>
<td>Aug17-2</td>
<td>0.19</td>
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4.4.1 Discussion of 8 mL/min Tests

Results of tests Aug18-8, Aug20-8A, and Aug20-8B are presented here, as each of these tests have all pressure sensors functioning (tests with pressure readings marked N/A in Table 4.1 had a broken sensor lead to the base pressure sensor). The remaining tests are described and discussed in Appendix 4.7. Figures 4.5–4.7 show the pore fluid pressure in the bed as a function of time, with the corresponding height time series obtained by image analysis. Pore fluid pressure is defined as the interstitial water pressure (e.g. Been and Sills, 1981), here given in excess of hydrostatic pressure; it is essentially the fluid pressure due to inability of the solid matrix to support its own self-weight. The pore pressures observed in Figures 4.5(a), 4.6, and 4.7(a) are similar in magnitude to those observed by Been and Sills (1981) during gravity sedimentation.
tests of a silty clay mud. Figure 4.8 shows the volume fraction of solids for tests Aug20-8A and Aug20-8B as functions of time, after filtering as previously described. The initial 0–0.21 spike at the beginning of the time series is a filtering artifact, due to the number of points in the series. The oven-measured initial volume fraction of solids for both tests was 0.19 (three replicates) prior to degassing, so it appears that a systematic error may exist in the gamma system calibration; in any case, the trend is towards a slightly lower final volume fraction of solids, but the change is not really measurable within the resolution of the current system. Figure 4.9 shows the pressure drop between the base sensor and the average value of the four bed sensors at each time instance (space/time average). In general, fluidization literature presents the pressure drop across the particle bed, similar to Figure 4.9. As each pressure differential (Δp) rises, the bed height rises concomitantly; a decrease in Δp is indicative of strong fluid flow near the sensed region (in this case, averaged over the entire bed). The initial step rise in each of the graphs of Figure 4.9 is caused by the start of the pump; this is more evident in Figures 4.5(a), 4.6, and 4.7(a), which show the actual pore pressure measurements. Measurements of a liquid-fluidized bed of coal particles by Asif et al. (1994) show a step decrease in Δp with increased flow through the bed. In general, the same trend is evident in the present study; although the flow rate remained constant, visual observations showed that the flow was localized in certain regions which correspond to a decrease in Δp measured by the sensor in that region. An overall increase in Δp is observed prior to cracking/fluidization of the bed, which is typically denoted by a cusp in the pressure trace, followed by a decrease in Δp as flow starts moving through the bed. The overall behavior of the bed is summarized thusly:
Figure 4.5: Pore fluid pressure and height of the bed as functions of time, for the Aug18-8 8 mL/min test.
Figure 4.6: Pore fluid pressure for each sensor, Aug20-8A 8 mL/min test (no images were taken for height measurement).
Figure 4.7: Pore fluid pressure and height of the bed as functions of time, for the Aug20-8B 8 mL/min test.
Figure 4.8: Volume fraction of solids as obtained by the gamma system plotted versus time for tests Aug20-8A and Aug20-8B, after low-pass filtering.
Figure 4.9: Pressure drop versus time for tests Aug18-8, Aug20-8A, and Aug20-8B, given in psi, using the average value of the four bed sensors.
1. Flow is initiated, $\Delta p$ decreases to some relatively constant value near 0.04 psi

2. The bed cracks, a cusp or short spike is evident in the pressure trace

3. Flow emanates from the crack or hole in the bed, and pore pressure decreases

Figure 4.10 shows the final bed state of test Aug18-8, with a closed crack that extended from sensor 4 along the side near sensor 5 and finally up to the bed surface between sensors 5 and 2. This crack closed shortly after a hole burst in the top of the bed (see the note on Figure 4.9(a)), and remained in the closed state through the end of the test. A scale of millimeters is provided in the figure. It should be reiterated that the $\Delta p$ vs. time graphs are averaged values of each sensor (2, 3, 4, 5) $\Delta p$ values, and that pressure events on a single trace can affect this average significantly; Figure 4.9(c) is a case in point, where cusps on sensor 2 (see Figure 4.7(a)) have appeared in the spatially averaged trace. Although no visible activity was observed near sensor 2 in test Aug20-8B, the peaks are above the typical noise level of the sensors. In this particular

Figure 4.10: Bed photograph showing the final state of test Aug18-8, scale in millimeters. The cracks are at the middle left of the photo.
case, most of the flow was emanating from the opposite side (near sensors 3 and 4) of the column, and it is likely that crack opening and closing that was not visible to the operators (to avoid exposure to the gamma radiation beam) near sensors 3 and 4 affected the pressure near sensor 2.

4.4.2 Overall Trends

Some variation in the base pressure is evident (Figure 4.11), but is essentially constant over the range of flowrates examined, with a slight increase in pressure with flowrate. A comparison of the value of $\Delta p$ when a crack was first observed as a function of the flow rate is shown graphically in Figure 4.12. There is significant scatter in the data, due to the somewhat subjective evaluation of the time at which cracking occurred, but the trend is evidently towards a lower $\Delta p$ for higher flowrates. It appears that $\Delta p$ approaches a limiting value of $\sim 0.04$ psi, which corresponds to the highest pore pressures in the bed itself.

Figure 4.11: Time-averaged pressure in the column base versus flowrate. Aug20-4 is excluded from the fit.
Figure 4.12: $\Delta p$ when a crack is observed as a function of volumetric flowrate. Aug20-4 is excluded from the fit.

It is possible that the lower flowrates allow more time for pressure to build in the system before cracking occurs, but further investigation is certainly warranted in this case.

Figure 4.13 shows the time average of the spatially-averaged value of the four bed pressure sensors over the duration of the test. Figure 4.13 indicates that the actual fluid pressure in the bed is negative at certain flowrates (below 4–6 mL/min). The only way that this could be possible in a pure fluid is if the energy due to fluid velocity at the sensor level is greater than the potential energy of the height change between the sensor level and the bed surface; however, this would require a high flow velocity and is clearly not the case. Other researchers have reported negative pressure in fluidized beds (Daw et al., 2000; Kage et al., 2000, e.g.), but it is unclear whether they performed a detrending operation on their data before presenting it (which would enforce a fluctuation about zero). Pore pressures less than that of hydrostatic pressure are observed in unsaturated soil due to matric suction (e.g. Simon and Collison,
2001), but this is not likely to be the case in the present study, due to the low volume of entrained air in the sediment bed. According to Muhunthan and Schofield (2000), a reduction in effective stress (unloading) may be caused by increasing pore water pressure while sediment particles are interlocked, as in the present case. Further, Muhunthan and Schofield (2000) note that a hydraulic gradient across the sediment during this stress reduction may cause a fluidization or fracture in the material, as observed in the present study, with similar crack or pipe cycling as well. This unstable state could also account for the variability in crack location and time of occurrence, at least partially.

### 4.5 Conclusions

The liquid fluidization behavior of kaolinite clay has been examined experimentally, with detailed measurements of pressure, density, and height. An incipient condition for fluidization was sought, and was defined for the pur-
poses of this paper to occur when cracking of the sediment bed was observed visually. The flowrates used for fluidization in these tests did not have a perceptible effect on the volume fraction of solids of the beds studied, which indicates that fluidization was far from total. Through numerous detailed experiments, it was shown that the behavior of the system results in a higher apparent strength (resistance to fluidization) at low flowrates (less than 4 mL/min). An incipient pressure drop for fluidization appears to exist at 0.04 psi, for the volume fraction of solids used in the present study. Several effects remain to be examined: effect of initial bed height; effect of gas flow; effect of higher flowrates. The next step recommended for this research is to focus on a particular flowrate (perhaps the 8 mL/min chosen here) and emphasize repeatability, as well as whether a possible long-term cyclical behavior exists.

4.6 Acknowledgments

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4.7 Appendix: Additional Test Results and Analysis

4.7.1 Test Aug12-2

The results of test Aug12-2 (2 mL/min) are presented in Figures 4.14–4.15. Due to a broken pressure sensor lead in sensor 1 (the base), no differential pressure readings are available. This test was characterized by an increase in bed height over time, with no large cracking in the side of the bed. The bed actually separated from the cylinder base as a piston ∼1200 s. into the test, after appearing to expand uniformly; this separation evidently allowed some flow to bypass the bed, as pressure then remained constant in the bed for the remainder of the test (Figure 4.14). The bed continued to rise, however, until surface cracks appeared in the top of the bed, at which point (∼3200 s.) upward movement of the bed ceased (Figure 4.15).

4.7.2 Test Aug12-4

The results of test Aug12-4 are presented in Figures 4.16–4.17. Due to a broken pressure sensor lead in sensor 1 (the base), no differential pressure readings are available. No abrupt pressure increase is visible in Figure 4.16 at 120 s. when the pump was turned on, which is unusual. Prior to 500 s., flow was observed moving up the side of the wall between sensors 5 and 2; at ∼840 s., a crack was observed near sensor 5, which was followed by a subsequent decrease in pressure near that sensor. It is likely that this crack occurred at 500 s., based on Figure 4.17, which shows an abrupt height change at this time. Adjacent sensors 2 and 4 also felt the decrease in pressure after this crack occurred, but sensor 3 did not. A second height change event occurred at 1400 s., and was
Figure 4.14: Pore fluid pressures for Aug12-2 test at 2 mL/min. No base pressure.

Figure 4.15: Height of the bed for the Aug12-2 test at 2 mL/min.
also followed by a decrease in the pore pressure of the bed.

4.7.3 Test Aug12-6

The results of test Aug12-6 are presented in Figures 4.18–4.19. Due to a broken pressure sensor lead in sensor 1 (the base), no differential pressure readings are available. The pump was started at 40 s., with a corresponding pressure change in sensors 2 and 5 almost immediately (Figure 4.18). A crack grew between sensors 4 and 5 (first noticed at 120 s., which corresponds with Figure 4.19), and appears to have expanded again at 200 s. before collapsing slightly (not recorded by the camera) at 600 s.. The overall bed state remained the same, and visible flow continued to come out of this crack, while pore fluid
pressure increased slightly. An event appears to have taken place near 900 s., but nothing was visible to the naked eye.

4.7.4 Test Aug13-8

The results of test Aug13-8 are presented in Figures 4.20–4.21. Due to a broken pressure sensor lead in sensor 1 (the base), no differential pressure readings are available. A large change in height is evident at 250 s., which corresponds with visual records of a crack opening in the side of the bed under sensor 4. No abrupt pressure change is evident at this point in time, although a cusp at 220 s. on the sensor 4 trace in Figure 4.20 suggests that the crack formed earlier; small pressure events are evident at 250 s. on sensors 2, 3, and 5, corresponding with the time that the crack was first observed with the naked eye.

4.7.5 Test Aug17-2

The results of test Aug17-2 are presented in Figures 4.22–4.23. No height information is available for this test, so the visual observations are the only other data available. Two outlets were used for this case, and all of the
Figure 4.18: Pore fluid pressures for Aug12-6 test at 6 mL/min. No base pressure.

Figure 4.19: Height of the bed for the Aug12-6 test at 6 mL/min.
Figure 4.20: Pore fluid pressures for Aug13-8 test at 8 mL/min. No base pressure.

Figure 4.21: Height of the bed for the Aug13-8 test at 8 mL/min.
remaining fluidization experiments. The pump was started at 240 s., and two cracks in the surface of the bed were visible by 1200 s., near the center of the column. It is likely that the small fluctuations evident in the sensor 3 trace of Figure 4.22 are due to this cracking. The flow of water from the top was low enough to make it difficult to determine the exact source of the flow, but it appeared to emanate steadily from a crack in the top nearest to sensors 2 and 3. The steady pressure is indicative of no major height changes in the system, as from a large horizontal crack in the sediment bed; separation from the column base, as in test Aug12-2, was not evident.
Figure 4.23: Pressure drop between the base and average value of the bed sensors for the Aug17-2 test at 2mL/min.

4.7.6 Test Aug18-2

The results of test Aug18-2 are presented in Figures 4.24–4.26. Although Figure 4.25 indicates a rather large drop in the bed height at 200s., there is little evidence of such a change in the pressure time series measurements. Overall, pressures remained fairly steady over the duration of this test, with some increase from the initial value; trends in sensor activity are similar to that in test Aug12-2, but show somewhat less activity than Aug17-2. In general, it appears at this point that low flowrates tend to reduce the intensity of pressure changes in the bed. A decrease in the differential pressure $\Delta p$ occurred at approximately the same time as a crack in the side of the bed (Figure 4.26), which in turn immediately preceded the height drop of Figure 4.25.
Figure 4.24: Pore fluid pressures for Aug18-2 test at 2 mL/min.

Figure 4.25: Height of the bed for the Aug18-2 test at 2 mL/min.
Figure 4.26: Pressure drop between the base and average value of the bed sensors for the Aug18-2 test at 2mL/min.
4.7.7 Test Aug18-4

The results of test Aug18-4 are presented in Figures 4.27–4.29. Approximately 190 s. into the test, a crack in the side of the bed was observed under sensor 4, extending towards sensor 5. Sensors 3 and 5 register some activity at this time (Figure 4.27), and the $\Delta p$ trace of Figure 4.29 indicates that a pressure release took place at about this time. Coincidentally, Figure 4.28 shows a step decrease in bed height at 200 s., indicating that some height change took place; the crack near sensor 4, however, did not close until $\sim 550$ s., according to the visual record of the test. As a crack closure is not listed in the visual record at 200 s., it is likely that it was due to a crack on the side opposite the viewers, or otherwise not visible.
Figure 4.28: Height of the bed for the Aug18-4 test at 4 mL/min.

Figure 4.29: Pressure drop between the base and average value of the bed sensors for the Aug18-4 test at 4 mL/min.
4.7.8 Test Aug18-6

The results of test Aug18-6 are presented in Figures 4.30–4.32. A crack formed between sensors 5 and 2 at 300 s., and a cusp in the height plot (Figure 4.31) is evident at that time. An increase in $\Delta p$ at 300 s. indicates a release of flow as the average pressure in the bed decreased; this decrease was strongest at sensor 2, according to the plot of pore pressure in Figure 4.30. The second crack noted in Figure 4.32 does not appear to have affected the pressure or height of the bed significantly; it appears that height increased gradually over the remainder of the test, as did the pore pressure.
Figure 4.31: Height of the bed for the Aug18-6 test at 6 mL/min.

Figure 4.32: Pressure drop between the base and average value of the bed sensors for the Aug18-6 test at 6 mL/min.
4.7.9 Test Aug18-10

The results of test Aug18-10 are presented in Figures 4.33–4.35. A 240 s., a 2 mm wide crack was observed near sensor 4, propagating towards sensor 5, which is typified by the small peak in the sensor 4 trace of Figure 4.33 at that time. Pressure near sensor 5 appears to have increased during that time, rather than decreasing as usually happens when a crack is near a sensor. It is possible that the crack focused flow towards sensor 5, rather than releasing flow from it (which would typically cause a pore pressure decrease). The only height change evident from the image analysis occurred at 230 s., so it is likely that the crack actually formed at the drop in height at 230 s.; up to that point, there had been a slight, gradual increase in bed height.
Figure 4.34: Height of the bed for the Aug18-10 test at 10 mL/min.

Figure 4.35: Pressure drop between the base and average value of the bed sensors for the Aug18-10 test at 10 mL/min.
4.7.10 Test Aug19-2

The results of test Aug19-2 are presented in Figures 4.36–4.39. The gamma radiation system was used to determine solids volume fraction for this test and the remaining tests discussed in this appendix. Figure 4.38 shows the sequence of events observed during the test; the only height change was at approximately 300 s., where the first crack was observed near sensor 4. Sensor 2 shows a sinusoidal variation in pressure with a peak-to-peak time of approximately 650 s.; this variation is also evident in sensor 3 with a lesser amplitude. Although no cracking or flow was observed near sensors 3 and 5, it is possible that they were influenced by height changes in other parts of the bed. Examination of Figure 4.36 reveals negative pore pressures at all of the sensors (pressure less than hydrostatic), indicating that some type of suction must be occurring in the bed. It is not likely that this is an instrument malfunction, as all sensors display similar values; however, the Aug12-2 and Aug17-2 tests do not exhibit negative pressure (and neither does the Aug12-4 test immediately following this one). The filtered volume fraction of solids plot (Figure 4.39) shows no overall trends, and particular events noted on corresponding pressure and height plots are likewise not evident.
Figure 4.36: Pore fluid pressures for Aug19-2 test at 2 mL/min.

Figure 4.37: Height of the bed for the Aug19-2 test at 2 mL/min.
Figure 4.38: Pressure drop between the base and average value of the bed sensors for the Aug19-2 test at 2 mL/min.

Figure 4.39: Volume fraction of solids for the Aug19-2 test at 2 mL/min.
4.7.11 Test Aug20-4

The results of test Aug20-4 are presented in Figures 4.40–4.43. Height changes in this test were large in comparison with other tests (Figure 4.41), with 1–2 mm spikes. Overall, the height and pressure (Figure 4.40) increased together, and the pressure traces are well-marked by cusps or inflection points at the time of a height change. From this test, it appears that pressure and height increase and decrease together, with respect to overall trends, and that large step increases in bed height are not permanent.

4.7.12 Test Aug20-6

The results of test Aug20-6 are presented in Figures 4.44–4.47. A crack near sensor 4 at 200 s. shows up as a small disturbance on the pressure trace...
Figure 4.41: Height of the bed for the Aug20-4 test at 4 mL/min.

Figure 4.42: Pressure drop between the base and average value of the bed sensors for the Aug20-4 test at 4 mL/min.

Figure 4.43: Volume fraction of solids for the Aug20-4 test at 4 mL/min.
(Figures 4.44 and 4.46), and a leveling off of the height increase (Figure 4.45). At 400 s., some activity is again evident, with a step pressure change at sensor 3; in this case, again, the pressure change was on the side opposite the visible cracking in the bed. Gradual increases were evident in the other sensors, until approximately 800 s., at which time pressure in the bed decreased again (Figure 4.44). As no significant height change accompanied this decrease in pore pressure, it seems that the bed fracture had already taken place, and flow was perhaps redistributing itself slightly. No indications of this activity are apparent in Figure 4.47, the volume fraction of solids.
Figure 4.45: Height of the bed for the Aug20-6 test at 6 mL/min.

Figure 4.46: Pressure drop between the base and average value of the bed sensors for the Aug20-6 test at 6 mL/min.

Figure 4.47: Volume fraction of solids for the Aug20-6 test at 6 mL/min.
4.7.13 Test Aug20-10

The results of test Aug20-10 are presented in Figures 4.48–4.51. The first event appears to have occurred at 200 s., as a small rise in the height curve is evident (Figure 4.49), concomitant with a change in pressure which is most evident in Figure 4.50. Pressure in the bed increased steadily on sensor 2 and sensor 5 (Figure 4.48), until 500 s.; a small dip in the height curve occurred at this time, and it is likely that the crack in the top which was noticed at approximately 600 s. formed at this time. Consistent with this assertion is the decrease in the bed pressure which began at 500 s.. The volume fraction of solids for this test (Figure 4.51) has an unusually large dip at 200 s., indicating that perhaps the higher flowrate of this test (10 mL/min) actually produced a detectable decrease in solids fraction; no other trend is evident, however.
Figure 4.49: Height of the bed for the Aug20-10 test at 10 mL/min.

Figure 4.50: Pressure drop between the base and average value of the bed sensors for the Aug20-10 test at 10 mL/min.

Figure 4.51: Volume fraction of solids for the Aug20-10 test at 10 mL/min.